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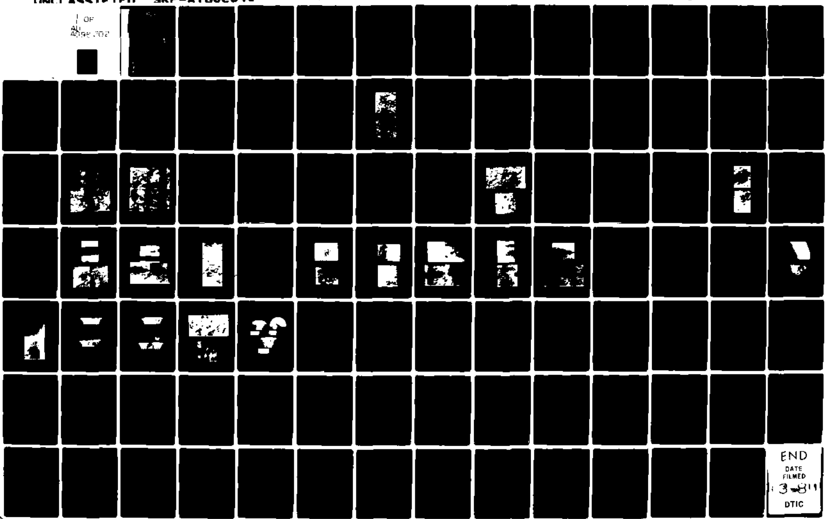

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FINAL REPORT
DRY LUBRICATION OF HIGH TEMPERATURE
SILICON NITRIDE ROLLING CONTACTS
N00019-79-C-0612

T. M. YONUSHONIS

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NOVEMBER 17, 1980

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Prepared for:

U.S. DEPARTMENT OF THE NAVY
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Favorable four ball test results indicate feasibility of silicon nitride bearings for high temperature turbine applications.

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Abstract

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Favorable four ball test results indicate feasibility of silicon nitride bearings for high temperature turbine applications.

Preface

This report presents results of an experimental study which investigated the solid lubrication of silicon nitride contacts. The work was conducted by SKF Technology Services, a division of SKF Industries, Inc., King of Prussia, Pennsylvania for U.S. Naval Air Systems Command, Washington, D.C. 20361 under contract number N00019-79-C-0612. This is the final technical report issued on the program which was conducted during the period extending from September 1979 to September 1980.

The silicon nitride ball processing and nondestructive evaluation for the programs "Dry Lubrication of High Temperature Silicon Nitride Rolling Contacts," NAVAIR Contract No. N00019-79-C-0612, and the "Engineering Design Data Program," NAVAIR Contract No. N00019-79-C-0148 were conducted concurrently. Therefore, basic materials data and NDE results were identical for both programs.

The author wishes to acknowledge the valuable discussions with A. Scilingo and J. Johnson. The contributions of F. Morrison, W. Rosenlieb, N. Miller, and D. Hahn in planning and conducting the experiments are greatly appreciated. The patient typing of this report by K. Helicher is also greatly appreciated.

1.0 Introduction:

Advanced turbine engines demand materials which operate at higher temperatures to meet performance and fuel economy goals. Government sponsored programs have been investigating high temperature alloys, complex cooling methods, and ceramic components for hot zone applications in advanced propulsion engines. These advanced engines will operate with higher temperatures at the bearing location. High bearing temperatures negate the use of conventional oil lubrication since the projected temperatures are well in excess of the oil flash point. These bearing locations will require development and use of solid lubricated, high temperature bearings. Bearing temperature capability will be a limiting factor in obtaining improved engine operating cycle efficiency.

SKF Industries, Inc. with funding from Naval Air Systems Command has been investigating [1, 2, 3] a bearing quality ceramic, hot pressed silicon nitride (HPSN), which has the potential to operate at the arduous service conditions projected by engine manufacturers. The purpose of the present program was to investigate existing solid lubricant systems that had potential for lubrication of silicon nitride at 1000°F (538°C) in air. The test program utilized a four ball test system to simulate bearing operation at high temperature.

2.0 Background:

Research into solid lubricant systems has revealed that no single solid lubricant is a panacea, and that each solid lubricant system has distinct advantages and limitations which must be considered prior to lubricant selection for screening tests and eventual application. A number of investigators have utilized solid lubricants to solve lubrication problems in specific gear, journal bearing, or rolling contact bearing applications where temperature requirements or vacuum considerations negated the use of liquid lubricants. Reviews of solid lubricant types, and general characteristics of solid lubricants have been provided in the literature [4, 5, 6, 7].

For the most part, solid lubricants such as graphite, MoS_2, WS_2, and CaF_2BaF_2 mixtures have been limited to reducing wear and galling of metals at low contact stresses. Haltner [8] has evaluated commercially available solid lubricant coatings for space shuttle journal bearings. His conclusion was that material wear could be significantly reduced by selection of the proper solid lubricant film provided that the design could tolerate a high coefficient of friction, $\mu_f \cong 0.1$ to 0.2.

Few researchers have investigated the solid lubrication of rolling contact bearings, although the use of solid lubricants such as graphite and MoS_2 in oils and grease is commonplace. Taylor and coauthors [9] functionally tested a ceramic rolling contact bearing from 1000°F (538°C) to 1500°F (815°C) with powdered MoS_2 supplied as the lubricant. In their tests, the powdered MoS_2 was introduced by a carrier gas which provided a protective atmosphere for the MoS_2. A nitrogen carrier gas was used at 1000°F (538°C)and an argon carrier gas was used at higher temperatures. Full scale bearing tests at 1500°F (815°C) had shown that operation at 8000 revolutions per minute was possible for 8 hours without significant bearing wear or damage.

Boes [10] has conducted full scale bearing tests at 1000°F (538°C) using titanium shrouded WSe_2-In-Ga cages as the lubricant source. The composite was transferred from the cage to the bearing surfaces. Successful operation of the bearing at 1000°F was limited to speeds below 30,000 RPM due to rapid wear of WSe_2-In-Ga composite. Rapid composite wear resulted in excessive transfer film build-up on races and balls causing loss of internal clearance and bearing seizure.

With NAVAIR sponsorship Norton Co. and SKF Industries, Inc. investigated solid lubrication of silicon nitride contacts in rolling four ball tests [3]. In this program, the silicon nitride was hot pressed to a net shape, then processed into 0.5 inch (12.7 mm) diameter grade 25 balls. Four ball tests at ambient and elevated temperatures indicated that silicon nitride could operate successfully with solid lubricant transfer films. However, test results from this program were complicated by three major factors which negatively biased the silicon nitride performance.

(1) The net shape silicon nitride produced was experimental and did not have the performance characteristics of Norton NC132 hot pressed silicon nitride in billet form.

(2) The AISI M1 tool steel cup contaminated the silicon nitride surfaces at the high temperature test conditions.

(3) The solid lubricant cage design was patterned from a conventional brass cage design for oil lubricated four ball tests. This cage design did not allow adequate transfer of graphite from the cage to the silicon nitride ball surface.

Although the test results from the previous NAVAIR program were biased by these three factors, the results still indicated

that the silicon nitride bearings were feasible for limited life applications. In addition, it was believed that the potential existed for long life bearings if a copious solid lubricant film was provided to the silicon nitride contacts.

3.0 Material Selection

3.1 Introduction

The ceramic material used for the manufacture of the 12.7 mm (0.500 inch) diameter balls and cups was Noralide NC132 hot pressed silicon nitride (HPSN) obtained from Norton Company. Bearing tests utilizing silicon nitride rolling elements made to this material specification have demonstrated oil lubricated fatigue lives comparable to M50 bearing steel [2].

Quality control measures were implemented in the areas of raw material inspection as well as non-destructive evaluation of finished parts to assure production of the highest quality silicon nitride balls. The effectiveness of the quality control procedures was manifested by uniform ball density, and minimal reject rate observed during fluorescent dye penetrant inspection of the finished balls.

The solid lubricant coatings and monolithic solid lubricant cage materials were selected based on availability, chemical compatibility with silicon nitride, ability to operate continuously at 1000°F (538°C), and the potential of decreasing the coefficient of friction between silicon nitride rolling contacts. The materials selected following the preceding criteria were graphite and WSe_2-In-Ga in monolithic compacts, and WS_2, MoS_2, and graphite as thin coatings. Other potential solid lubricant systems were not used since they did not meet one or all of the selection criteria. For example, CaF_2-BaF_2 eutectics have been demonstrated in journal bearing applications [11]. However, the CaF_2-BaF_2 composition has a high coefficient of friction at 1000°F (538°C) and similar fluoride systems have been effective silicon nitride etchants [12].

In addition to decreasing the silicon nitride wear rate by solid lubrication, enhancement of the silicon nitride surface by nitrogen ion implantation and nitriding was considered.

3.2 Silicon Nitride

3.2.1 Norton Certification

The Norton certification data of powder lot HN-15 and the billets used in this program are presented in Appendix A. Quality control procedures at Norton consist of analyzing the powder

lot for key elements, checking individual billets for flexure strength at room temperature and 2500°F (1370°C), structural consistency using x-ray radiographic techniques, and density. In addition to these measurements, proprietary processing parameters are retained and analyzed by Norton. All billets used in this program exceeded the minimum requirements for Noralide NC132 hot pressed silicon nitride(HPSN).

3.2.2 SKF Material Certification

SKF Technology Services augmented the Norton quality control procedures with additional x-ray radiographic inspection, fracture surface analysis, optical analysis, density and hardness measurements. These additional evaluations were selected because they were believed to yield the maximum amount of information on billet quality with a minimum cost and schedule impact.

3.2.2.1 X-Ray Radiography A 150 mm x 15 mm x 3 mm slab was sliced from each HPSN billet received from Norton for this program. These sections were x-ray radiographed to check for possible segregation or x-ray density variations in planes parallel to the hot pressing direction. One silicon nitride billet had x-ray density variations. This billet was replaced by the Norton Co. and was not used for ball manufacture.

3.2.2.2 Fracture Surface and Ceramographic Analysis

Fracture surfaces were analyzed by the scanning electron microscope (SEM). Polished HPSN sections were analyzed optically by Nomarskii interference contrast. Fracture surfaces from billet to billet were, for all practical purposes, identical. Both intergranular and transgranular fracture modes were observed. This fracture behavior is typical of HPSN. In general, the HPSN was a fine grained, dense, uniform material as analyzed by these techniques. Microscopic analysis of the polished sections was unable to distinguish between inclusion content from billet to billet. However, low power and visual observations revealed slight differences in the material's polishing behavior. For example, one billet may have a darker overall coloration or may polish with fewer silicon nitride grain pullouts.

In summary, low power optical analysis indicated subtle billet to billet variations in the HPSN. However, detailed SEM and optical microscope analysis confirmed that all billets used in this program were similar and acceptable for further processing.

3.2.2.3 Hardness and Density Vicker's or diamond pyramid hardness was measured at a 500 gram load. These hardness measurements were taken to provide a relative ranking of the HPSN

billets. As can be seen in Table 1, slight differences in hardness from billet to billet were observed, but these variations are not believed to be significant. All of the hardness values exceeded the minimum value of 1400 kg/mm^2 specified by SKF.

Density of thirty-four finished balls, selected at random, was calculated from ball dimensions and the dry weight. Average density of the HPSN balls was 3.25 g/cm^3, Table 2, which is in good agreement with Norton's certification data. The measured density of each ball exceeds SKF's minimum requirement of 3.2 g/cm^3. This is an improvement compared to balls made from near-net shape material. Thirteen percent of the near-net shape hot pressed silicon nitride balls checked on a previous NAVAIR Load Life Program [1] had densities lower than 3.2 g/cm^3.

3.3 Cage Materials

3.3.1 Graphite

Tests conducted in the NAVAIR program entitled, "Severe Environment Testing of Silicon Nitride Rolling Elements," [3] have demonstrated that graphite solid films transferred from a cage to rolling contact surfaces resulted in high temperature silicon nitride wear rates comparable to oil lubricated bearing steels. Due to the apparent success of graphite at high temperature, three vendors were selected that manufacture graphites capable of sustained 1000°F (538°C) operation.

The graphite materials and vendors selected were as follows:

AFX-5QE	-	POCO Graphite, Inc.
P2003 Graphite	-	Pure Carbon Co.
56HT Graphite	-	Pure Carbon Co.
CJPS Carbon Graphite	-	Union Carbide Corp.

Cages were machined from each of these four materials for evaluation in this program.

3.3.2 WSe_2-In-Ga Composite

The Westinghouse Corp. compact, WSe_2-In-Ga composite, has been reported by Boes [10] to be an excellent solid lubricant for metal bearings up to 1000°F (538°C). Static oxidation of compacted WSe_2-In-Ga composites is negligible to 1400°F (760°C). Cages were fabricated from WSe_2-In-Ga for evaluation.

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Table 1

Vicker's Hardness Data Measured on Silicon Nitride Billets

Billet Number	Hardness (kg/mm^2)	Billet Number	Hardness (kg/mm^2)
1280	1648	1292	1514
	1679		1545
	1679		1545
1281	1576	1293	1545
	1545		1545
	1545		1644
1282	1545	1294	1610
	1610		1610
	1610		1679
1284	1679	1295	1576
	1610		1545
	1610		1616
1285	1610	1296	1679
	1610		1679
	1644		1610
		1297	1679
			1610
			1610

average hardness = 1607 kg/mm^2; 33 data points

standard deviation = 51 kg/mm^2

Table 2

Bulk Density of 34 Finished Silicon Nitride Balls

Density (g/cm^3)	Density (g/cm^3)
3.249	3.243
3.252	3.252
3.250	3.249
3.247	3.243
3.250	3.254
3.252	3.249
3.245	3.249
3.251	3.249
3.240	3.250
3.247	3.269
3.249	3.266
3.237	3.250
3.237	3.247
3.270	3.240
3.252	3.252
3.251	3.239
3.250	3.251

average density = 3.25 g/cm^3; 34 data points

standard deviation = 0.008 g/cm^3

3.4 Solid Lubricant Coatings and Surface Modifications

3.4.1 Solid Lubricant Coatings

Suppliers of solid film coatings were solicited for candidate solid lubricant materials having potential for operation at 1000°F (538°C) and 400 ksi (2760 MPa) contact stress. Because of the limited number of silicon nitride cups available in this program, only the spindle and support balls were coated by the solid films. It was theorized that the low contact stress at the ball-race interface, approximately 85 ksi (590 MPa), minimized the need for the coating on the race surface.

A WS_2 coating was applied to finished silicon nitride balls by Dicronite of Rotary Components, Inc. E/M Lubricants supplied two solid lubricant coatings. A Microseal 100-1 graphite coating and a MoS_2 coating in an inorganic binder were applied by E/M Lubricants.

3.4.2 Surface Modifications

Gazza [13] has shown that a nitriding treatment applied to hot pressed silicon nitride containing Y_2O_3 additions can eliminate catastrophic oxidation of that material. Nitriding may also improve the oxidation resistance of hot pressed silicon nitride containing an MgO based hot pressing aid.

Two silicon nitride spindle balls were nitrided by Mr. Rorabaugh at Garrett using a fourteen day reaction bonding cycle.

Ion implantation has been used to improve the wear resistance of metals. Dr. J. Hirvonen of NRL was contacted for ion implantation of silicon nitride ball surfaces. Recent work conducted at NRL on improving silicon nitride wear resistance was unsuccessful [14]. Therefore, ion nitriding was not pursued during this program.

4.0 Component Design, Manufacture and Inspection

4.1 Silicon Nitride Balls

4.1.1 Dimensional Quality

All balls used in this program conformed to AFBMA grade 25 quality requirements which had been specified for the test. Grade 25 balls have a maximum allowable 2 point out of roundness of 25 x 10^{-6} in (0.64 μm) and a maximum surface roughness of 1 x 10^{-6} in AA (0.02 μm AA).

4.1.2 Nondestructive Evaluation

The silicon nitride balls used in this program were subjected to a 100% fluorescent dye penetrant inspection. A sensitive fluorescent dye penetrant (Zyglo ZL30; ZR-10A) was used to impregnate the balls at Peabody Testing/Magnaflux. Visual inspection of the entire ball surface was conducted using ultraviolet light. The improved material quality of balls manufactured from billets as compared to near net shape silicon nitride was readily apparent [1]. Historically, fluorescent penetrant inspection of near net shape silicon nitride balls yielded a material related defect rate of 1 defect per 2.8 in^2 (18.1 cm^2) of silicon nitride surface area inspected. The billet material inspected during this program had a defect rate of 1 defect per 190 in^2 (1226 cm^2) of silicon nitride inspected. Thus, the rejection rate of balls manufactured from billet material was one-sixtieth of that observed by SKF on near-net shape silicon nitride.

Figure 1 presents scanning electron micrographs of the final polished silicon nitride balls. No evidence of interconnected porosity was observed on the ball surfaces during examination in the scanning electron microscope. Residual grinding marks visible at 2500x magnification do not affect the silicon nitride ball performance.

4.2 Silicon Nitride Cups

4.2.1 Design

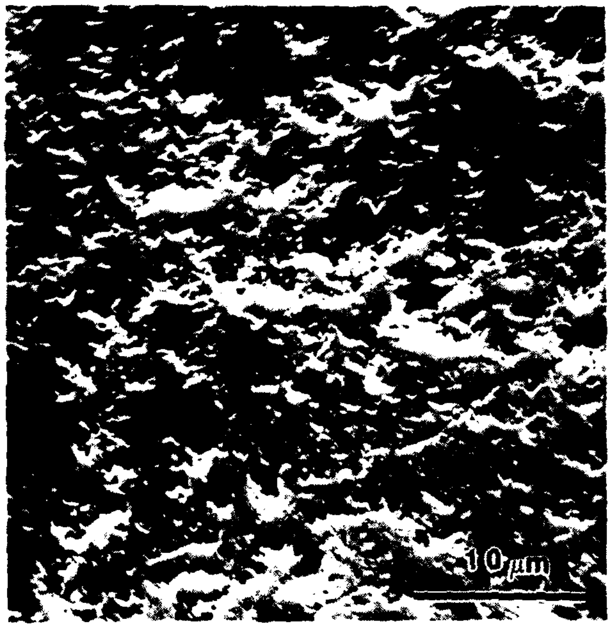
The silicon nitride cup design is shown in Figure 2. A 44° contact angle was chosen to allow testing with 3 support balls plus a spindle ball (four ball test) with a solid lubricant cage separating the support balls. This design also allows the cage to be removed and a fourth support ball inserted into the cup (five ball test). By removing the cage, it is possible to investigate the effects of solid lubricant coatings or unlubricated contacts independent of the presence and function of a cage.

Selection of silicon nitride for the cup minimized contamination at high temperatures. Previous tests at SKF utilizing an AISI M1 tool steel cup revealed that the tool steel would grossly contaminate the silicon nitride contacts at 1000°F (538°C) [3]. Eliminating the tool steel cup removed the possibility of metal contamination.

4.2.2 Dimensional Quality

A summary of SKF's metrological evaluation of five of the six cups made for this program is presented in Table 3. Although one cup's race was 0.0002 inch (0.005 mm) oversize on the diameter, this cup was considered acceptable to use in these tests. Four silicon nitride cups were within SKF's limits as specified by SKF Drawing No. L23777-1. The sixth cup was only used for the system check out.

Figure 1 Scanning electron micrographs of the typical surface finish from two different silicon nitride balls.



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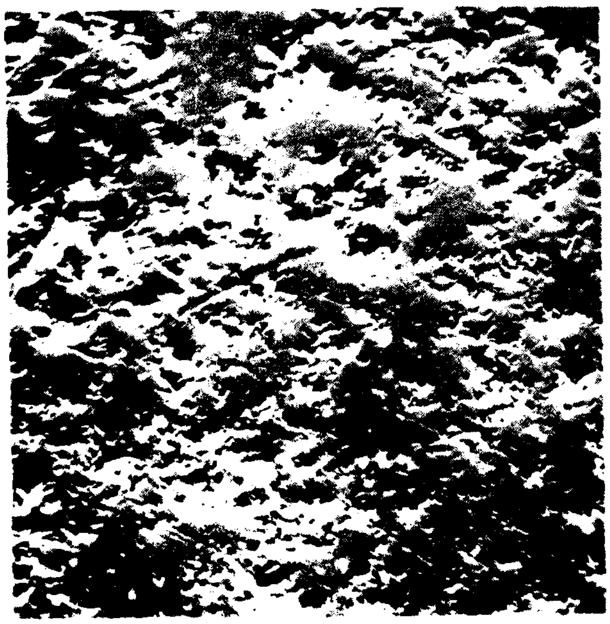

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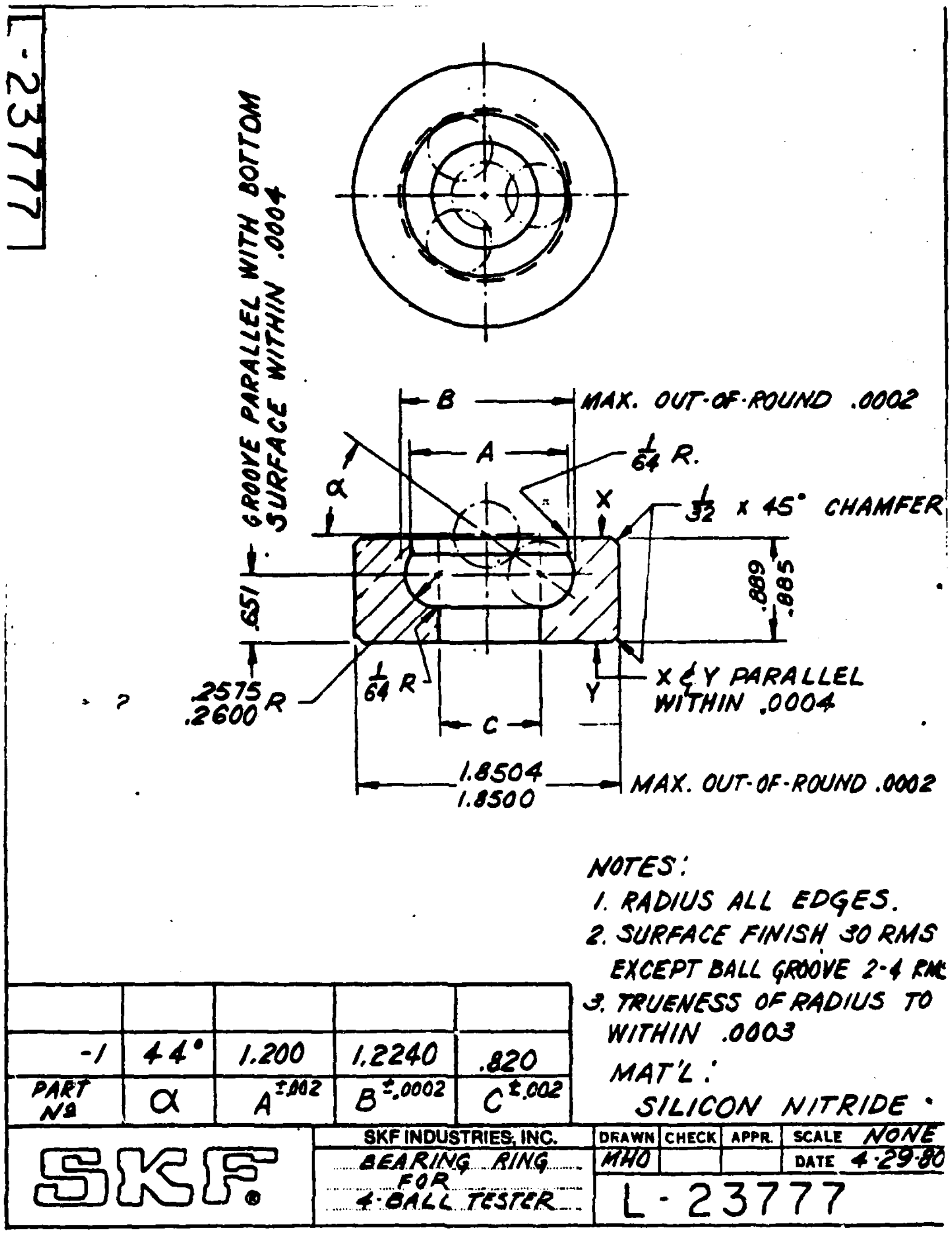
Figure 2 44° Contact Angle Silicon Nitride Cup



ALL DIMENSIONS ARE IN INCHES.

TABLE 3

Dimensions and Surface Finishes of Silicon Nitride Cups

Silicon Nitride Cup Number		Diameter at 0° in (mm)	Diameter at 90° in (mm)	Surface Finish μin (μm)	Maximum Out of Round μin (μm)	Groove Parallel with Base μin (μm)
1		1.2241 (31.092)	1.2240 (31.090)	1.49 (0.038)	94 (2.4)	118 (3)
2		1.2240 (31.090)	1.2240 (31.090)	1.27 (0.032)	79 (2.0)	196 (5)
3		1.2239 (31.087)	1.2240 (31.090)	1.29 (0.033)	63 (1.6)	236 (6)
4		1.2239 (31.087)	1.2242 (31.095)	1.79 (0.045)	47 (1.2)	157 (4)
5		1.2242 (31.095)	1.2244 (31.100)	2.80 (0.071)	157 (4.0)	118 (3)
Specified by Print L-23777-1	max.	1.2242 (31.095)	1.2242 (31.095)			
	min.	1.2238 (31.085)	1.2238 (31.085)	≤4 (≤0.1)	≤200 (≤5)	≤400 (≤10)

4.3 Cages

4.3.1 Design

A cage design compatible with the silicon nitride cup design is presented in Figure 3. Cage pockets for the silicon nitride balls were designed to permit transfer of the solid lubricant from the cage surface to the contact area between the support balls and the spindle ball. Transfer of the solid lubricant film into this area would occur even during single axis rotation, i.e., tracking of the support balls in the silicon nitride cup. Without a proper cage design it would be possible to have the silicon nitride contacts unlubricated during tracking.

4.3.2 Manufacture and Inspection

The solid lubricant cages were manufactured to the SKF design by the material vendors or their designated subcontractor. Cages were visually inspected at SKF Industries. Dimensional acceptability of the cages was determined by inserting the cages in the silicon nitride cup with the three silicon nitride support balls in place and observing clearances and contact areas. Dimensional quality of the cages was sufficient to allow evaluation of all solid lubricant materials.

5.0 Experimental Procedure

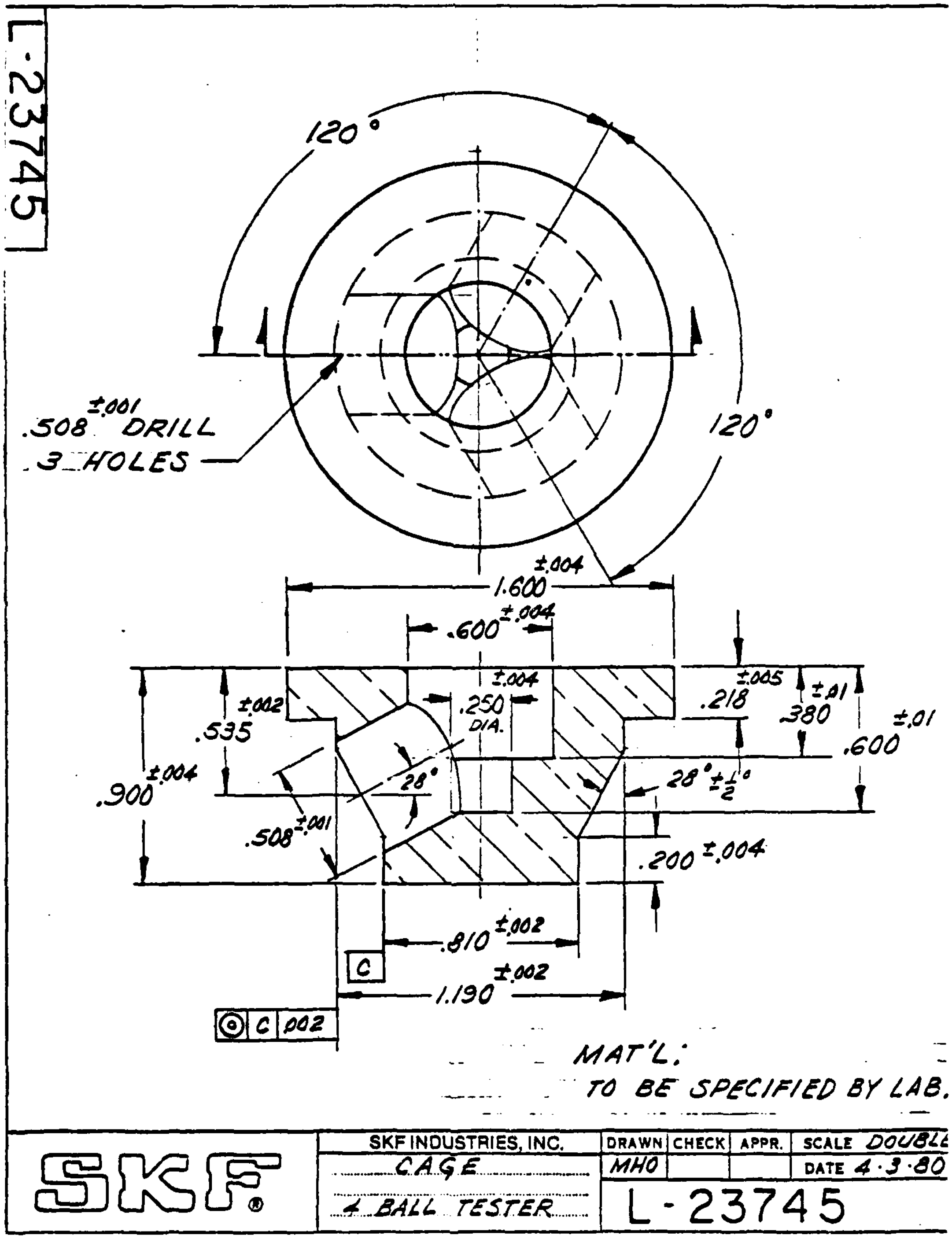
5.1 Test Equipment

Two rolling four ball test systems were used to study solid lubrication of silicon nitride rolling contacts. A schematic of the four ball test system is shown in Figure 4. The metal housing has integral heaters capable of 1000°F (538°C) operation for extended periods of time. In the four ball test, the spindle ball is dead weight loaded through a vertical arbor against three support balls which orbit the spindle ball in a stationary race. The spindle ball is fixed in a cone shaped seat containing a spring loaded arbor. The spindle ball geometry is shown in Figure 5. The three support balls are positioned in a silicon nitride cup 120° apart by a solid lubricant cage. The positioning of the support balls insures similar Hertzian stresses at each spindle ball - support ball contact.

The silicon nitride cup containing the support balls, spindle ball and cage is piloted in a metal housing by a finger-like arrangement, which positions the inner diameter surface of the cup. This mechanism assures positive positioning of the cup during the thermal cycling which normally occurs during a test.

The four ball tests were conducted at 10,000 rpm (1047 rad/sec). This four ball test system is equipped with a vibration

Figure 3   Solid Lubricant Cage for Four Ball Tests



ALL DIMENSIONS ARE IN INCHES.

Figure 4 Schematic drawing of high temperature rolling four ball test system.

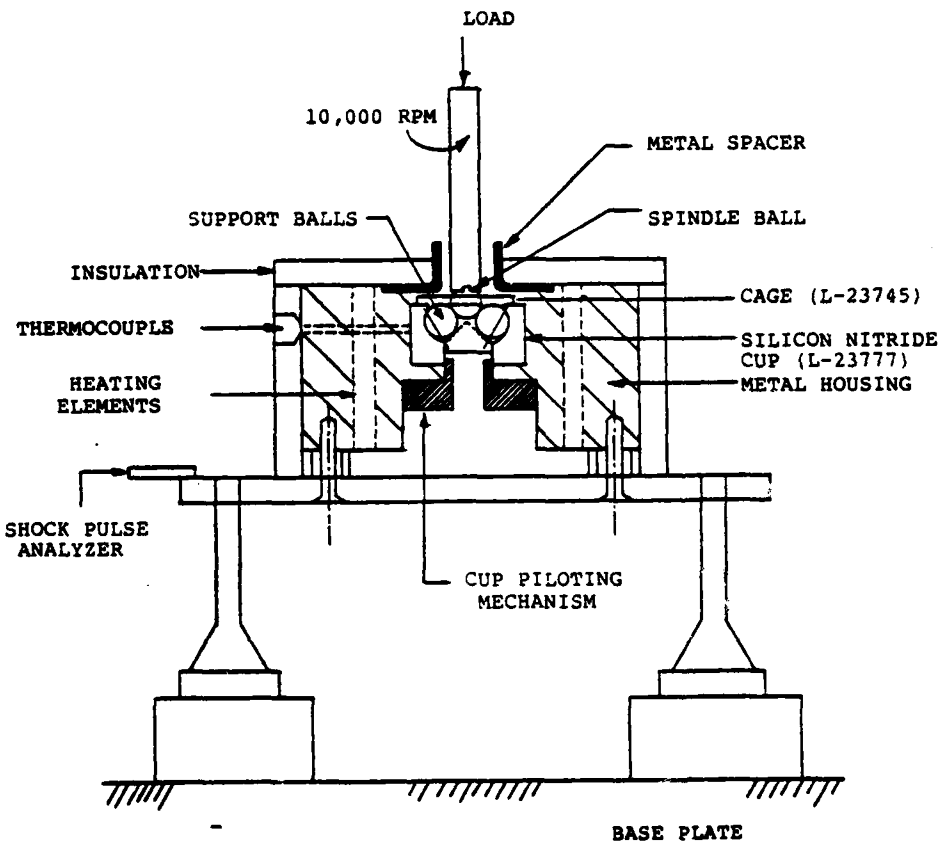
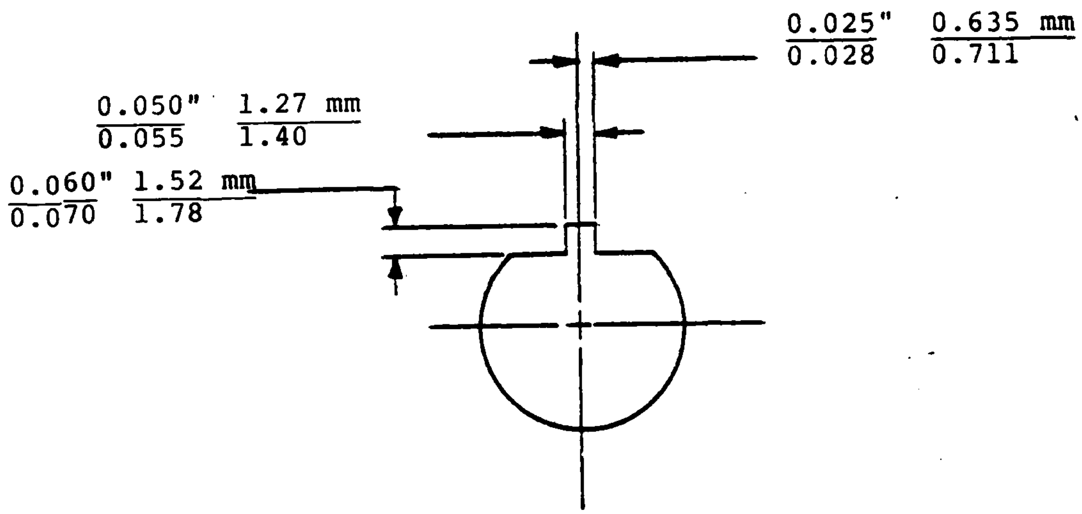


Figure 5 Silicon Nitride Spindle Ball



sensitive shut-off transducer. An increase in the vibration level is indicative of spalling fatigue failure or extreme wear of the spindle ball. When a vibration increase is sensed by the transducer, the test machine is shut down automatically to prevent progression of damage.

The Hertzian contact stress at the rolling contacts between balls is controlled by system geometry and the load applied through the arbor. A calculation of the load required for a 400 ksi (2760 MPa) contact stress is presented in Appendix B.

5.2 Test Procedure

A silicon nitride cup was inserted into the metal housing by preheating the housing to 300°F (150°C). The cups were cleaned by a naptha soaked cloth and visually examined prior to each test. The silicon nitride cups in this program were used for multiple test runs.

The silicon nitride balls were ultrasonically cleaned in acetone prior to testing. Initial weight of each silicon nitride ball was determined after drying at 300°F (150°C) for three hours.

Graphite and WSe_2-In-Ga cages were evaluated in the condition as supplied by the manufacturer. The cages were retained in a desiccator until test evaluation.

Silicon nitride support balls and cages were placed in the cup. A spindle ball was placed in the graphite cage and contacted the three support balls. The load was then applied to the spindle ball through a stainless steel spindle which mated to the flat ground on the spindle ball. The spindle was rotated by hand to assure proper seating of the four ball test assembly. When the operator was satisfied that the assembly was properly seated, an electric drive motor was switched on. The spindle ball speed reached 10,000 rpm in seconds. Temperature was then increased to the desired level, usually 1000°F (538°C). After the temperature reached equilibrium, approximately 30 minutes, testing continued until either the vibration switch actuated due to failure or planned test duration was completed.

5.3 Characterization of Solid Lubricants

The graphite based materials were analyzed by emission spectroscopy, powder x-ray diffraction, and scanning electron microscopy to determine differences, if any, among the various grades of graphite selected. W. B. Coleman Co. Testing Laboratories conducted the spectrographic analysis. Powder x-ray diffraction was conducted at SKF Industries, Inc., using chromium $K\alpha$ radiation at a scanning rate of 1° 2θ/minute. Fracture surface analysis was

done at SKF on an ETEC Autoscan SEM with attached x-ray wavelength analyzer.

The WSe_2-In-Ga composite material was analyzed by scanning electron microscopy and powder x-ray diffraction.

5.4 Wear Monitoring

Silicon nitride wear volumes and wear coefficients were estimated by analysis of diamond stylus traces obtained at three locations situated 120° apart on each spindle ball. These traces, perpendicular to the wear track, were used to determine the volume of silicon nitride removed. Wear volumes were determined by calculating the maximum material removed based on geometrical considerations of track width and stylus trace deviation from a true ball surface. The wear coefficients represent the maximum wear rate expected. Wear coefficients were calculated using the maximum width of the wear track obtained from the three stylus traces. A wear coefficient for each test was calculated using the following equation [15, 16]:

$$\text{wear coefficient:} \quad = \quad k \quad = \quad 0.129 \frac{pV}{SCa^3\sigma}$$

where

p = hardness, 1500 kg/mm^2

V = volume removed

S = spin to roll ratio, 0.64

C = stress cycles

a = contact radius, 33×10^{-3} inch (0.083 mm)

σ = maximum compressive stress, 400 ksi (2760 MPa)

6.0 Results and Discussion

6.1 Characterization of Cage Materials

Scanning electron photomicrographs of graphite and WSe_2-In-Ga fracture surfaces are shown in Figures 6 and 7. Fracture surfaces were used to define the material microstructures. As can be seen in these figures, a wide variety of microstructures existed among the various graphite materials and the WSe_2-In-Ga composite. The POCO AFX-5QE graphite had a fine uniform microstructure consisting of equiaxed 5 µm graphite grains. Two graphite grades, Pure Carbon P2003 and Union Carbide CJPS, had similar microstructures. Both materials contained flat graphite platelets

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Figure 6 Fracture Surface ... 2063 and 56HT Grade Graphite ... Electron Photomicrographs.

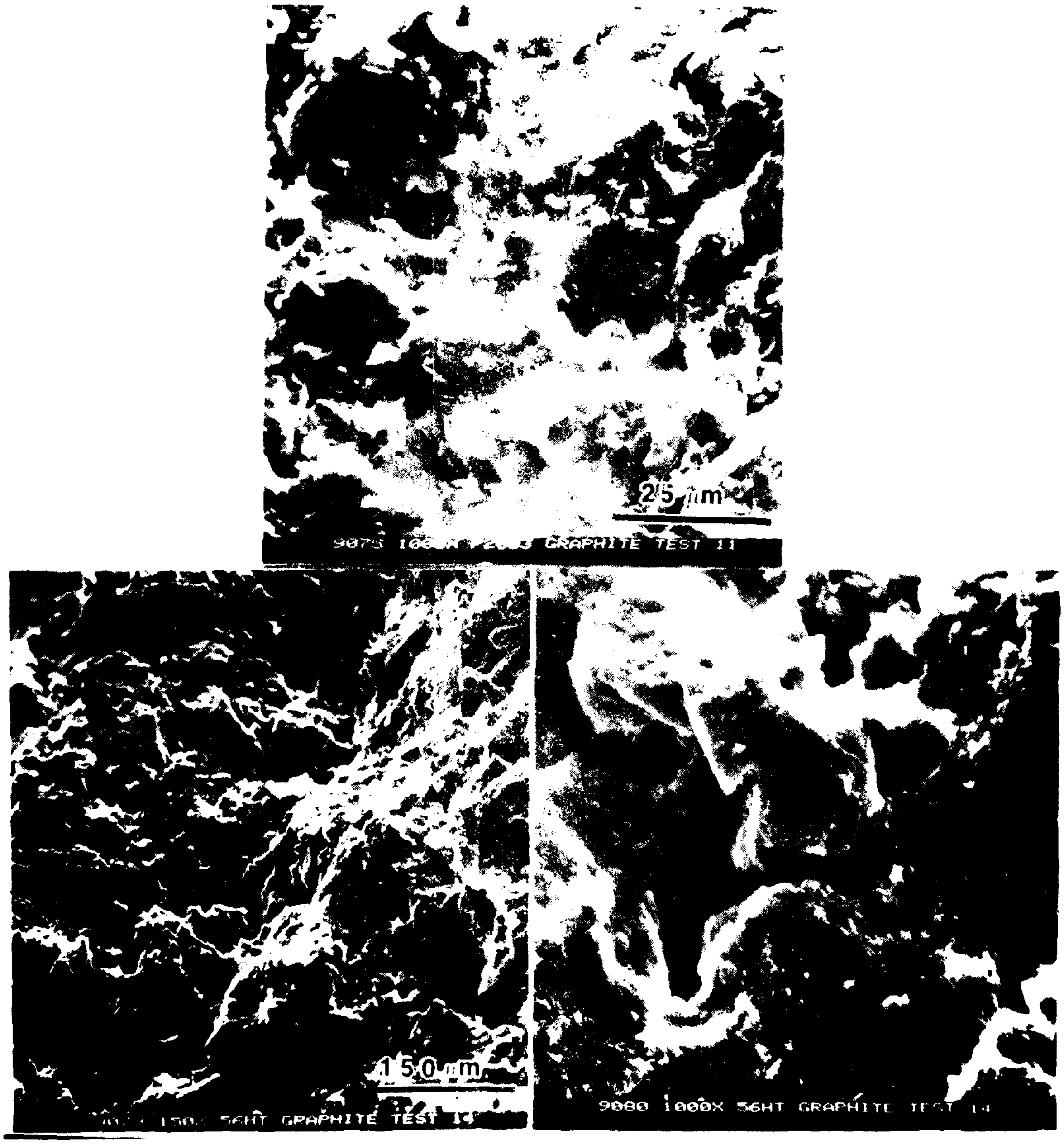
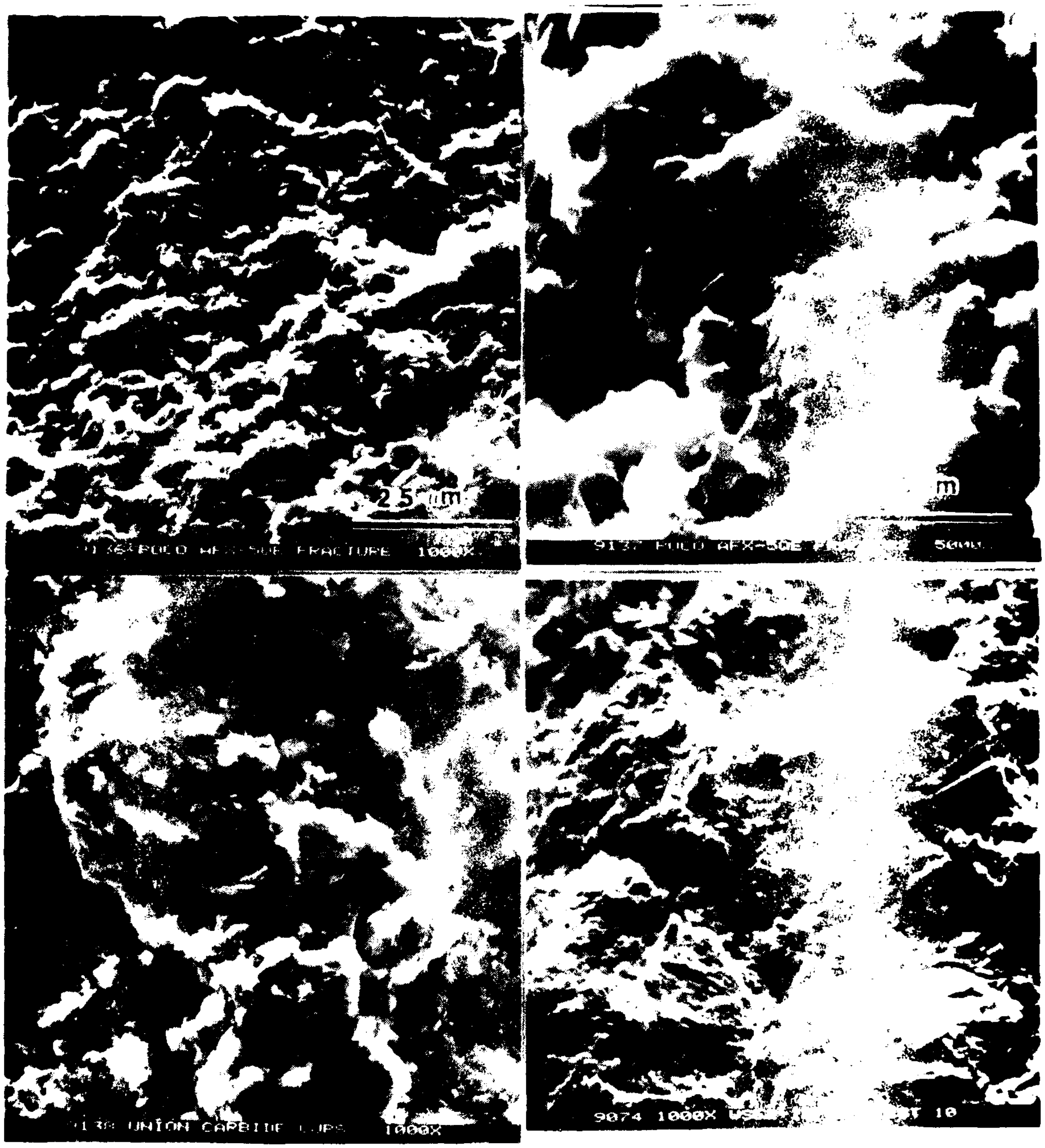


Figure [illegible] Fracture surface [illegible] Graphite [illegible]
Union Carbide CJ[illegible] graphite, [illegible]
Westinghouse Cor[illegible]
Scanning Electron [illegible] micrographs.



and had similar visual appearances. The microstructure of these materials was not as uniform as the POCO Graphite AFX-5QE. Another graphite examined, Pure Carbon 56HT, had a large grained structure. As can be seen in Figure 6, large pores were readily observed between the graphite grains. The only nongraphite material investigated in monolithic form was the WSe_2-In-Ga composite. This material had a bimodal grain size distribution. Ten to twenty micron WSe_2 particles were surrounded by a matrix of fine particles, Figure 7.

Powder x-ray diffraction of the graphite cages revealed some startling similarities between the high temperature graphite grades evaluated. Table 4 presents a summary of the powder x-ray diffraction results. As noted in Table 4, graphite was the major phase in the graphite cages. By characterizing the low intensity peaks, other similarities in the graphites were noted. For example, unindexed peaks at d-spacings of approximately 6.10 and 5.30 Å indicate that similar impregnants were used in POCO Graphite AFX-5QE, and Pure Carbon materials. The Union Carbide CJPS graphite appeared to contain a different impregnant based on x-ray diffraction results and emission spectrographic analysis.

X-ray diffraction of the WSe_2-In-Ga composite revealed WSe_2, and a Ga/In/Se phase. X-ray diffraction peak heights were similar to those observed by other researchers [18].

Spectographic results of graphite cages obtained using an exterior surface for a target are shown in Table 5. POCO Graphite AFX-5QE and Pure Carbon 56HT graphites contained comparable quantities of the same elements. Both materials had additives based on zinc and phosphorus, possibly combined as a zinc phosphate. Pure Carbon's P2003 material had an impregnant containing zinc, magnesium and phosphorus, while the Union Carbide CJPS appeared to have an impregnant consisting of aluminum phosphates.

6.2 Rolling Element Tests

6.2.1 Introduction

Appendix C presents a sequential listing of summaries of rolling element tests conducted during the course of this program. Section 6.2 contains a synopsis of the rolling contact tests segregated according to lubricant types and temperatures. The best silicon nitride performance was obtained using graphite cages. Results obtained at 1000°F (538°C) with graphite cages are presented in Section 6.2.3.3.

6.2.2 Initial Rolling Contact Tests

The first rolling contact tests were conducted using the five ball configuration without any solid lubrication. A zir-

Table 4

Powder X-Ray Diffraction of Cages After 1000°F (538°C) Exposure

Cage Type	Phases Identified	Additional d-Spacings (Å) Not Identified
POCO Graphite AFX-5QE	graphite (major)	6.09, 5.31, 5.05, 3.17, 3.11 (minor peaks)
Pure Carbon Co. 56HT	graphite (major)	6.14, 5.29, 4.26 (minor peaks)
Pure Carbon Co. P2003	graphite (major)	6.09, 5.28, 4.75, 3.09, 2.98, 2.91, 2.82 (minor peaks)
Union Carbide CJPS	graphite (major)	5.25, 4.09 (AlP04?), 2.51 (AlP04?)
Westinghouse Corp. WSe_2-In-Ga	WSe_2, W, and Ga/In/Se phase	

Table 5

Spectographic Analysis of Pure Carbon Co. 56HT and P2003 Graphites, Union Carbide CJPS Carbon - Graphite, and POCO Graphite AFX-5QE Graphite.

	56HT	P2003	CJPS	AFX-5QE
Ag	0.000X(H)	0.0X	0.000X	0.000X
Al	0.00X	0.X	MINOR(L)	0.00X
As	NF	NF	NF	NF
Au	NF	NF	NF	NF
B	0.00X	0.00X	0.00X	0.00X
Ba	NF	NF	NF	NF
Be	NF	NF	NF	NF
Bi	NF	NF	NF	NF
Ca	0.0X	0.X	0.0X	0.0X
Cb	NF	NF	NF	NF
Cd	NF	NF	NF	NF
Co	NF	NF	NF	NF
Cr	0.00X	0.0X	0.00X	0.00X
Cu	0.00X	0.X	0.00X	0.0X
Fe	0.0X	0.X	0.0X	0.0X
Ga	NF	NF	NF	NF
Ge	NF	NF	NF	NF
Hg				
In				
Ir				
K	NF	NF	NF	NF
Mg	0.00X	MINOR	0.00X	0.00X
Mn	0.000X	0.000X	NF	0.000X
Mo	0.00X	NF	NF	NF
Na	NF	NF	NF	NF
Ni	0.00X	0.0X	0.00X	0.0X

	56HT	P2003	CJPS	AFX-5QE
P	MINOR	MAJOR(L)	MAJOR(L)	MINOR
Pb	NF	0.0X	NF	NF
Pd				
Pt				
Rh				
Ru				
Sb	NF	NF	NF	NF
Si	0.0X	MINOR	MINOR	0.0X
Sn	NF	NF	NF	NF
Sr	NF	NF	NF	NF
Te	NF	NF	NF	NF
Ti	0.0X	0.00X	0.00X	0.00X
V	NF	NF	NF	0.0X
W	NF	NF	NF	NF
Zn	0.X(H)	0.X	NF	0.X(H)
Zr	NF	NF	NF	NF

KEY:		%
MAJOR	=	OVER 5%
MINOR	=	1 to 5%
.X	=	.1 - .9%
.0X	=	.01 - .09%
.00X	=	.001 - .009%
.000X	=	.0001 - .0009%
N. F.	=	NOT FOUND

REMARKS:
(H)=high, (L)=low.

conia spindle was used to drive the spindle ball. A zirconia spindle was chosen since the material has a low thermal conductivity. The zirconia spindle was found to result in test difficulties due to zirconia debris generated and spindle run out. Although the unloaded spindle run out was held to less than 0.001 in (0.25 mm) problems were encountered with alignment. Test results revealed that the zirconia spindle was inadequate due to two major factors: (1) zirconia debris contaminated the bearing, (2) spindle run out increased to 0.007 in (0.18 mm) when loaded. The zirconia spindle was replaced with a stainless steel tapered metal spindle. The stainless steel spindle maintained the same run out, held to within 0.0015 in (0.038 mm), in the loaded and unloaded condition. The stainless steel eliminated the alignment problem by eliminating the untapered ceramic-metal interface. This spindle also removed the problem of zirconia debris contaminating the silicon nitride bearing.

To eliminate the possibility of contamination from alumina fiber insulation surrounding the metal housing a small, metal, chimney-like arrangement was used to separate the insulation from the test cavity.

After correcting problems resulting from alignment and abrasive contamination, a test (Test 3) was initiated using silicon nitride balls without any lubrication film. It was theorized that these tests would establish a baseline for dry operation of silicon nitride and determine the ability of the oxide surface layer to protect the silicon nitride surface at high temperature. Oxide surfaces are known to result in a considerable lowering of the coefficient of friction in metal systems [19].

In test 3, the temperature was increased from ambient to 1000°F (538°C) over a 27 minute period. The temperature remained at 1000°F (538°C) for approximately four minutes. Extreme wear of the spindle ball, support balls, and cup was observed after disassembly of the full complement silicon nitride bearing without solid lubrication. The silicon nitride spindle ball weight loss was 0.376 grams or eleven percent of its total mass. Wear of the spindle ball resulted in greater conformity of the spindle ball - support ball contacts resulting in a considerable decrease in the maximum compressive stress. A considerably greater weight loss would have been observed if the contact pressure had been constant throughout the test.

Also noted was a fine, tan powdery debris coating the entire five ball assembly. A powder x-ray diffraction pattern of the debris disclosed that the powder was β-silicon nitride. Peak broadening indicated that the silicon nitride powder was extremely fine grained and possibly contained a deformed crystal structure. Photomicrographs of the powder verified that the powder

contained many particles submicron in size. Scanning electron photomicrographs of the silicon nitride spindle ball and debris are shown in Figure 8.

The results from this test dramatically demonstrate the necessity of lubrication for high temperature silicon nitride rolling contacts. Solid lubrication is required to realize low wear rates and long life for bearings at 1000°F (538°C). Due to the establishment of an extremely disappointing baseline for silicon nitride in an unlubricated system, the program plan was altered. Initially, duplicate baseline silicon nitride wear rates were to be determined without solid lubrication. Later, tests would evaluate solid lubricant coatings, and solid lubricant cages supplying a transfer film to the silicon nitride balls. Evaluation of silicon nitride without lubrication at high temperature revealed a low probability of success for thin solid lubricant coatings; therefore, existing solid lubricant coatings were matched with compatible solid lubricant cages in an effort to significantly improve results.

6.2.3 1000°F (538°C) Tests

6.2.3.1 Solid Lubricant Coatings

Two solid lubricant coatings, tungsten disulphide and graphite, were tested in conjunction with cages manufactured from solid lubricants. It was decided to utilize a cage in addition to the WS_2 and graphite solid lubricant coatings due to the severe wear observed during evaluation of the silicon nitride in the unlubricated condition. In an attempt to maintain chemical compatibility, Dicronite's WS_2 coating was evaluated with a Westinghouse Corporation WSe_2-In-Ga cage and E/M Lubricants Microseal 100-1 graphite coating was evaluated with Pure Carbon Co. 56HT and P2003 graphite cages.

Four ball test noise was significantly reduced when a Dicronite WS_2 coating applied to Si_3N_4 balls was evaluated with WSe_2-In-Ga cage during Tests 4 and 5. However, the noise generated during these tests increased as a function of time. This noise increase was believed to be associated with the attrition of the WS_2 coating on the silicon nitride balls. Visual and SEM examinations of the silicon nitride balls revealed that the coating was removed from all contacting surfaces in the thirty minute test.

E/M Lubricants Microseal 100-1 graphite coating did not reduce silicon nitride wear when evaluated in conjunction with the Pure Carbon 56HT and P2003 graphite cages. This result was partially due to the superior performance of the graphite based cages compared to the WSe_2-In-Ga cages. Evidently, the graphite coating hindered the solid lubricant transfer from the

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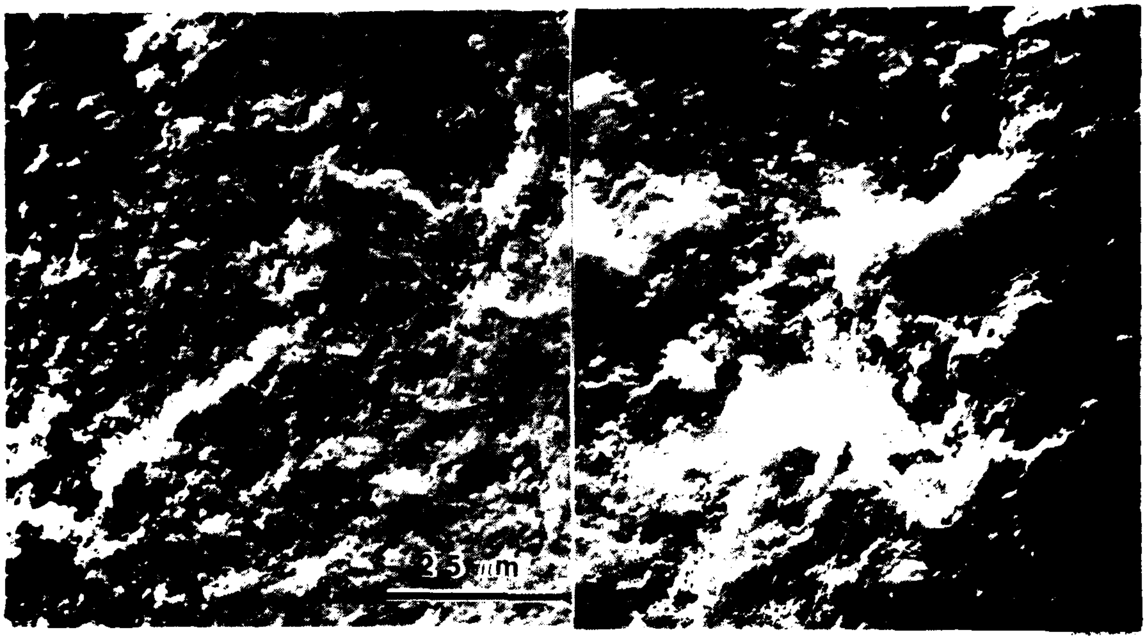
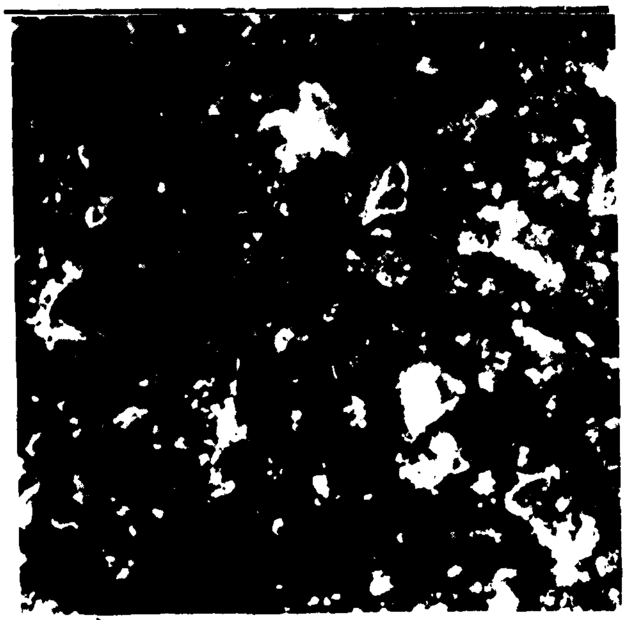
Figure 8 Scanning electron [illegible] of the silicon spindle ball surface [illegible] silicon nitride debris observed when [illegible] lubrication was not available to the rolling contact.
Test Conditions: 1000°F (538°C) for 4 minutes.



8976 1000x 8978 2500x

A. Silicon Nitride Surface



8930

B. Powdery Debris

SKF [illegible]

cage to the ball surface. Table 6 compares results of tests with and without the solid lubricant coatings. As can be seen in this table, the silicon nitride wear coefficients with solid lubricant coatings were approximately an order of magnitude greater than the wear coefficients determined without the graphite coating on the silicon nitride balls.

Another E/M Lubricant coating, MoS_2, was evaluated without a cage in a five ball test. The spindle and support balls had a 0.0025 to 0.005 inch (0.06 to 0.012 mm) thick coating. Test 25 ran smoothly from room temperature to 970°F (520°C). At the 970°F test temperature, an increase in vibration and noise was noted. The test was stopped after 25 minutes had elapsed. Visual inspection revealed that the MoS_2 coating had worn off the ball surfaces and that considerable wear of the spindle ball had occurred.

The solid lubricant coatings investigated, WS_2, MoS_2, and graphite, would not be acceptable for practical high speed, high temperature silicon nitride bearings. The expected life of a bearing protected by these coatings would be measured in seconds in the 1000°F temperature range. The coatings did not decrease the silicon nitride wear rate when used in conjunction with a monolithic solid lubricant cage. In fact, the graphite coating seemed to hinder the performance of the best graphite based cage evaluated, Pure Carbon Co.'s P2003 graphite. Judicious selection and matching of solid lubricant coatings with solid lubricant cages may eventually assist bearing start up. For example, the WS_2 coating appeared to assist the silicon nitride run in when tested with a WSe_2-In-Ga cage. Development of solid lubricant coatings compatible with the Pure Carbon P2003 graphite may be beneficial at lowering the starting torque of a complete silicon nitride bearing. Further investigations in this area would be required if a protective solid lubricant coating is deemed necessary for satisfactory bearing operation.

6.2.3.2 Westinghouse WSe_2-In-Ga Composite

The WSe_2-In-Ga composite was evaluated in Tests 4 and 5 in conjunction with a WS_2 coating. Tests 10 and 28 were conducted without the WS_2 coatings on the silicon nitride balls. In Tests 4, 5, and 10, the WSe_2-In-Ga composite did not provide a stable solid transfer film for silicon nitride. Based on the noise generated during these tests, the WSe_2-In-Ga composite appears to offer marginal lubrication films for silicon nitride rolling contacts. In Test 10, the drive motor was switched on after the test system had equilibrated at 1000°F. Normally, the rolling four ball test is initiated at room temperature, then the temperature is increased to 1000°F. During Test 10 a considerable amount of vapor was observed emanating from the test apparatus. The test was conducted at 10,000 rpm for a total of five minutes;

Table 6

Wear Rates of Silicon Nitride With and Without Solid Lubricant Coatings

Test	Cage/Ball Coating	Temp. (°F)	Hours	Stress Cycles ($x10^6$)	Wear Volume (mm^3)	Wear Coefficient ($x10^{-7}$)
4	WSe_2-In-Ga/WS_2	to 950	0.5	0.72	0.034	890
5	WSe_2-In-Ga/WS_2	to 650	0.28	0.38	0.003	150
6	P2003/100-1 Graphite	1000	34	46	0.168	69
7	56HT/100-1 Graphite	1000	40	54	2.59	900
25	None/MoS_2	to 970	0.42	0.56	0.538	18,300
9	P2003/None	1000	61	82	0.03	7
8	56HT/None	1000	50.4	68	0.61	170

then the test was stopped by the operator. Rig disassembly revealed that the WSe_2-In-Ga composite cage had fractured in a plane parallel to the plane containing the outer ring race. Considerable WSe_2-In-Ga solid lubricant material coated surfaces in contact with the cage and support balls. Figure 9 shows optical micrographs of the heavy WSe_2-In-Ga buildup on the spindle ball. Similar lubricant build up was observed on the support balls and cup. The copious WSe_2-In-Ga solid lubricant film transfer was beleived to decrease the cage pocket - support ball clearance, resulting in a wedging effect which fractured the cage.

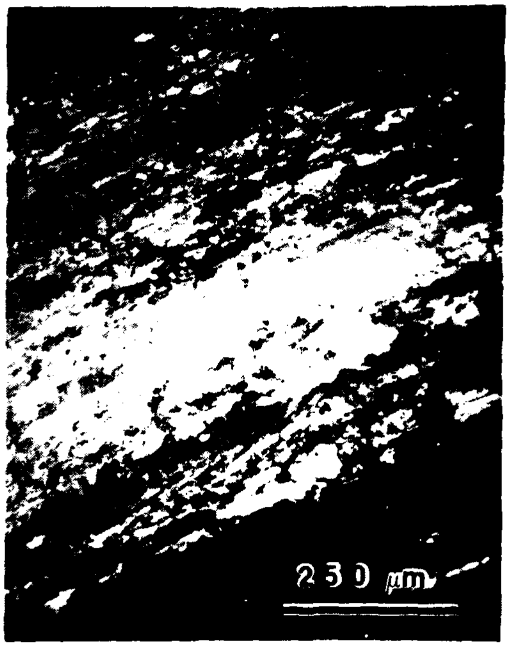
A final test of the Westinghouse WSe_2-In-Ga composite, Test 28, was conducted while monitoring the kinetic energy dissipated by the bearing as described in Paragraph 6.4 with the shock pulse analyzer. A sharp rise in the energy monitored was noted in the temperature range 400°F (200°C) to 750°F (400°C), indicating a possible breakdown of the WSe_2-In-Ga cage. Disassembly of the test apparatus revealed that the cage was fractured similar to the cage in Test 10.

In summary, the WSe_2-In-Ga composite as it now exists is not an acceptable high temperature solid lubricant for silicon nitride rolling contacts. The material did not have adequate strength to survive ball-cage impacts at high temperature. Evidently, large particles of WSe_2-In-Ga are removed from the cage surface, crushed, and transferred to the silicon nitride ball surface. The cage instability of the WSe_2-In-Ga composite at high temperature in a dynamic environment would eliminate WSe_2-In-Ga from consideration for high speed, high temperature bearings. It is believed that considerable refinements in retaining and applying this solid lubricant to the contact zone will be required to achieve the reproducibility required of a solid lubricant cage material. In addition to the stability problem, the high density of metals W, Ga, and In, would complicate high speed, solid lubricant bearing design.

6.2.3.3 Graphite

Table 7 contains a summary of the silicon nitride wear rates obtained at 1000°F (538°C) using graphite cages. Pure Carbon Co. P2003 graphite resulted in the lowest silicon nitride wear rates. Wear coefficients were approximately two orders of magnitude less than the wear coefficients obtained at high temperature during the previous program [3]. The success of the graphite cages in decreasing the silicon nitride wear rate was believed to be due to a combination of factors. First of all, the hot pressed silicon nitride used in the present program had improved wear resistance compared to net shape silicon nitride. This observation was noted during the ball lapping operations. The cage design was also believed to significantly improve the results. The cage design shown in Figure 3 allowed the graphite transfer

Figure 9 Optical micrographs of Si_3N_4 spindle ball showing build up of WSe_2-In-Ga composite during Test 10. A Westinghouse Corp. WSe_2-In-Ga cage was used as the solid lubricant source. Test conditions: approximately 5 minutes at 1000°F (538°C).



100x

400x

Table 7

Summary of Silicon Nitride Wear Rates at 1000°F Obtained with "New" Graphite Cages

Test	Cage	Hours	Stress Cycles ($x10^6$)	Wear Volume (mm^3)	Wear Coefficient ($x10^{-7}$)
9	P2003	61	82	0.03	7
11	P2003	258	348	0.07	4
12	P2003	139	187	0.02	2
13	P2003	256	345	0.04	2
8	56HT	50.4	68	0.61	170
14	56HT	130.7	176	0.41	44
15	AFX-5QE	50.5	68	0.35	98
18	AFX-5QE	71.2	96	0.145	29
16	CJPS	49.2	66	0.29	85
19	CJPS	25.3	34	0.55	309

film to lubricate the spindle ball contact area. The cage design used in the previous program [3] was ineffective at supplying a graphite transfer film to the contact area.

All graphite cages evaluated transferred lubricant films to the silicon nitride support balls in a very short time. Although the actual time required to transfer the graphite to the test ball was not measured, it was believed to occur in seconds. Because of initial rapid film transfer, smooth four ball test operation was experienced when graphite cages were used. This result was contrary to test operation observed with a WSe_2-In-Ga composite lubricant source.

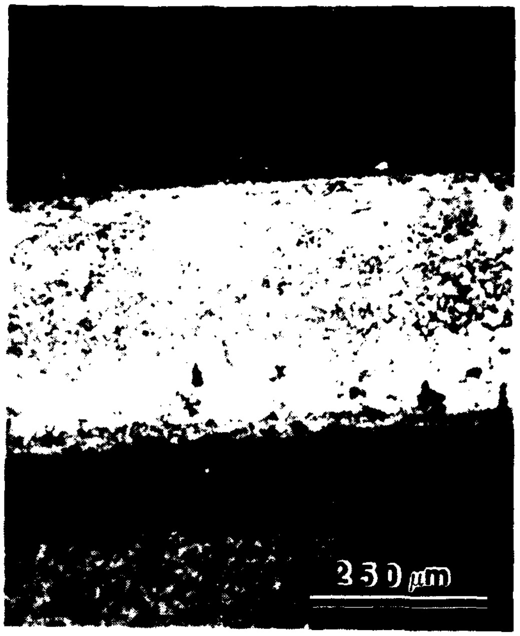
As stated previously, the Pure Carbon P2003 graphite resulted in extremely low silicon nitride wear coefficients. In fact, during long term tests, the silicon nitride wear rate may have decreased with increasing numbers of stress cycles. Two factors could be responsible for decreased wear. First of all, the contact stress decreases due to the wear of the silicon nitride spindle ball because the contact area increases slightly. Calculations have shown that the increase in contact area would decrease the contact stress by 50 ksi (345 mPa). It was unlikely that the small decrease in contact stress was significant. A second factor, the establishment of a stable solid lubricant film, would be expected to result in a decrease in wear as a function of time. Inspection of the support balls revealed that the balls were tracking in the silicon nitride cup. Bands of solid lubricant had formed on the support balls. Stable transfer of the solid lubricant from the support balls to the contact area during longer term tests was probably responsible for the slight decrease in wear coefficients at longer test times.

Figures 10 and 11 show optical and SEM photomicrographs of typical silicon nitride wear tracks which experienced P2003 graphite lubrication. Figure 10 shows an optical micrograph of the silicon nitride surface. Note that the solid lubricant film was not continuous. A continuous solid lubricant coating was apparently not necessary to decrease the silicon nitride wear rates.

Scanning electron photomicrographs, Figure 11, also show a fragmented film. The solid lubricant film morphology was not as well defined in this test. It should be noted that silicon nitride surface distress was not observed on silicon nitride balls tested with new Pure Carbon Co. P2003 graphite cages.

Support balls were also examined by optical and SEM methods. Figure 12 shows SEM photomicrographs of a partial solid lubricant coating on the support ball surface. X-ray wavelength spectroscopy was unsuccessfully used in an attempt to determine the solid lubricant film composition. This result was not totally

Figure 10 Optical micrographs of Si_3N_4 spindle ball wear track from Test 9. A Pure Carbon Co. P2003 graphite cage was used as the solid lubricant source. Test conditions: 1000°F (538°C) for 60.9 hours (82 million stress cycles).



Overall view of wear track 100x

Edge of wear track 500x

Center of wear track 500x

Figure 11 SEM photomicrograph of Si_3N_4 spindle ball wear track from Test 12. A Pure Carbon Co. P2003 graphite cage was used as the solid lubricant source. Test conditions: 1000°F (538°C) for 139 hours (187 million stress cycles).

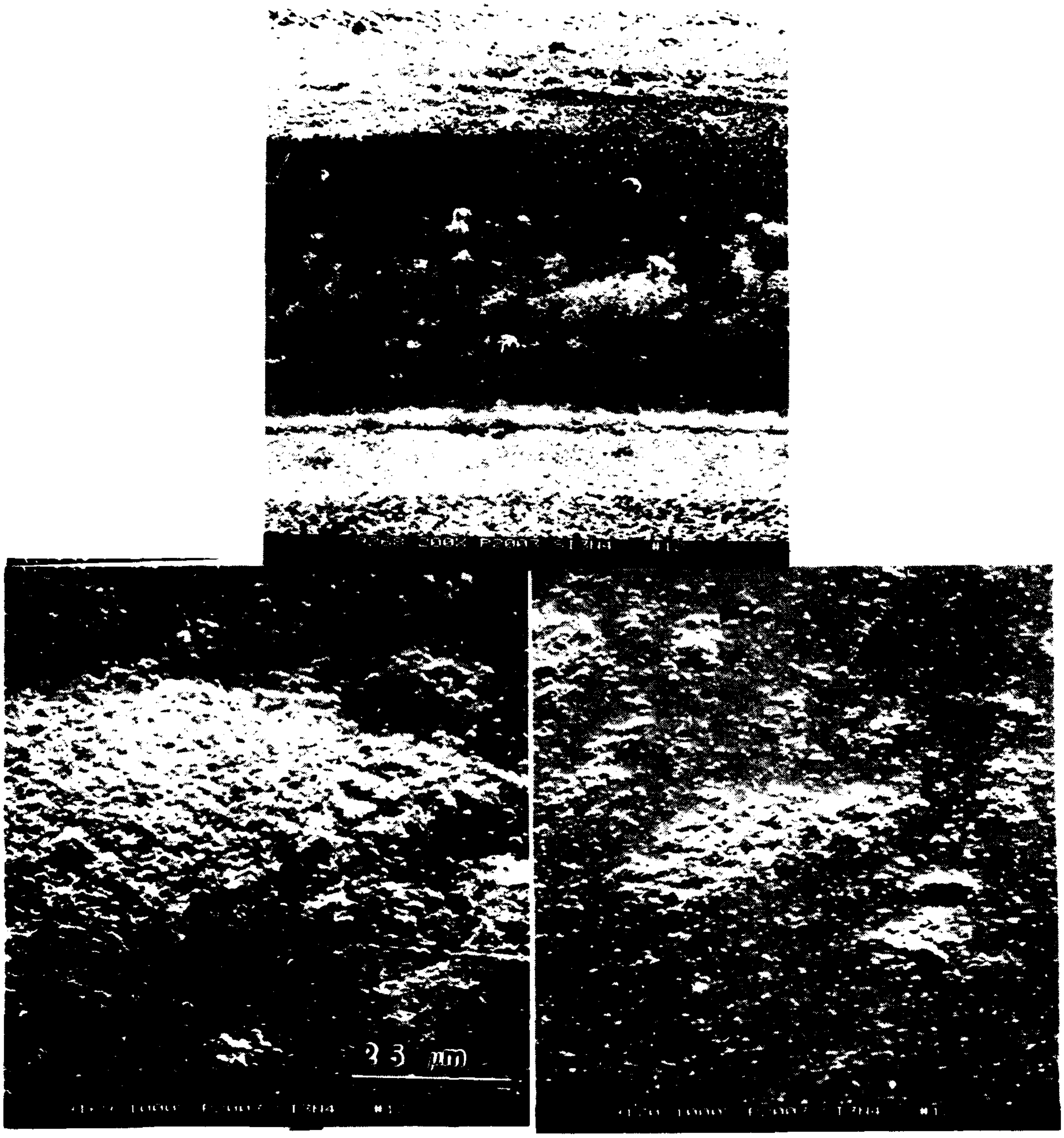
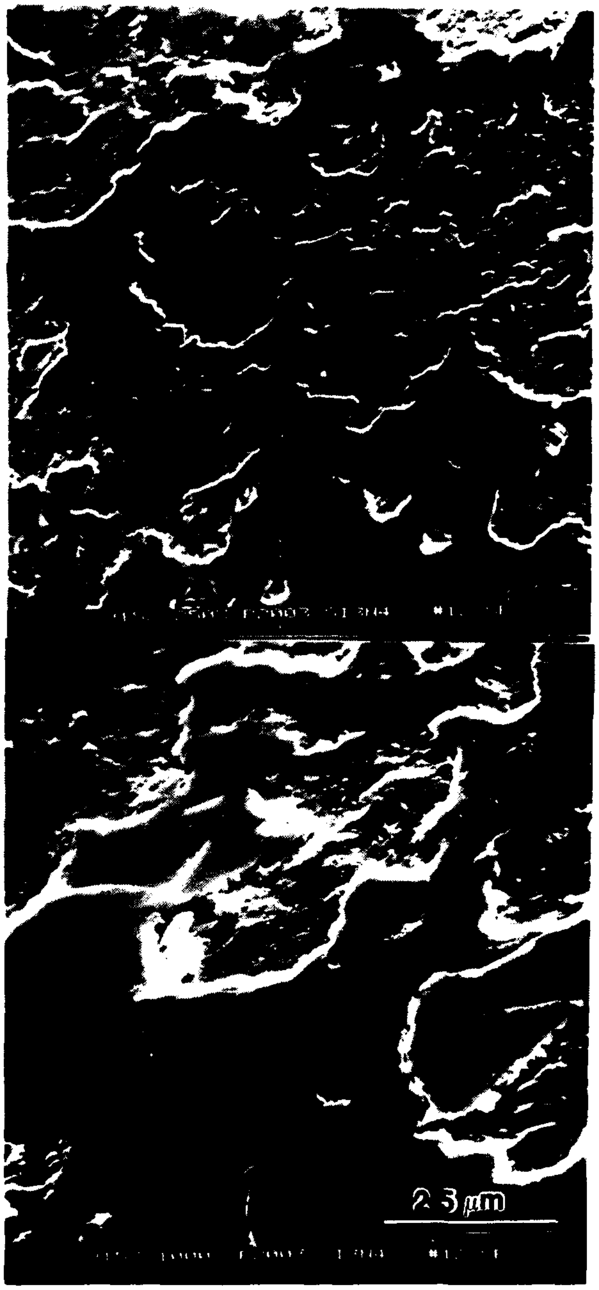


Figure 12 Scanning electron photomicrographs of solid lubricant coating on support ball. Lubricant was supplied by a Pure Carbon P2003 graphite cage. Test Conditions: 1000°F (538°C) for 139 hours (187 million stress cycles).



unexpected since thin carbon films are very difficult to detect by this technique. Scanning auger microscopy would probably be successful at positively identifying the film composition.

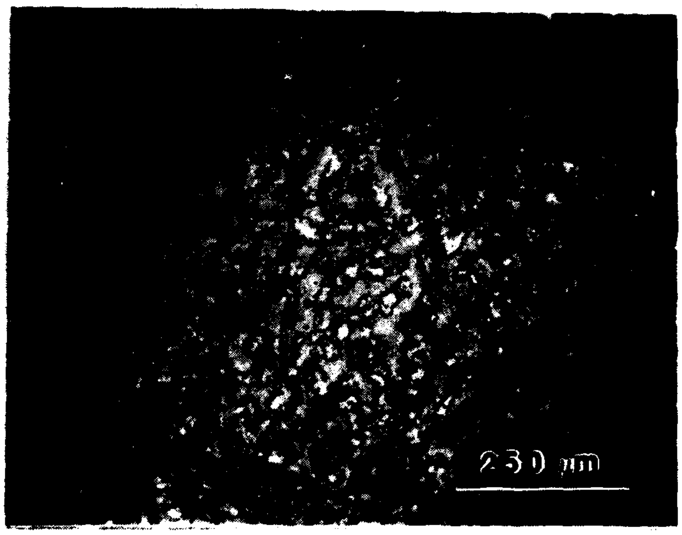
Considerably greater silicon nitride wear rates were observed when Pure Carbon 56HT, POCO Graphite AFX-5QE, and Union Carbide CJPS materials were used as the solid lubricant source. As can be seen in Table 7, the wear coefficient obtained with these materials was approximately one to two orders of magnitude greater than silicon nitride wear coefficients obtained with a Pure Carbon P2003 cage. Of considerable interest is the fact that graphite microstructure had no obvious influence on the results. For example, Pure Carbon P2003 graphite and the Union Carbide CJPS graphite had similar microstructures, but different impregnants. The Pure Carbon P2003 graphite produced effective solid lubricant transfer films while the Union Carbide CJPS was not effective. Figure 13 shows optical micrographs of support balls with a heavy build up of a film from the CJPS graphite cage. Even though a considerable build up of graphite based solid lubricant transfer films was here observed with the CJPS graphite, this film was not successful in decreasing wear of silicon nitride.

Two other graphite materials, Pure Carbon 56HT and POCO Graphite AFX-5QE, had completely different microstructures. POCO graphite AFX-5QE had the finest grain size and possessed a uniform microstructure. The Pure Carbon 56HT material had a coarse microstructure. It should be noted that both materials had similar impregnants in the zinc phosphate system. In view of the similar impregnants it should not be surprising that the wear coefficients for silicon nitride tested with Pure Carbon 56HT and POCO Graphite were similar. Apparently, the graphite microstructure has a minimal impact on lubricating ability for silicon nitride contacts, but the impregnant significantly alters the performance of the solid lubricant film.

Figures 14, 15, 16, and 17 represent typical silicon nitride wear tracks which were lubricated by the Union Carbide CJPS, Pure Carbon 56HT, and POCO Graphite AFX-5QE material. It is evident that the silicon nitride surface contained microfissures in the wear track. Optical and SEM analysis indicated that the microfissures did penetrate the silicon nitride, i.e., the microfissures were not confined to the thickness of the solid lubricant transferred film. The microfissures observed in poorly lubricated silicon nitride contacts are similar to those observed from sliding wear of metals [20].

In summary, significant differences in silicon nitride wear coefficients and degree of surface distress were observed while using graphite cages as a solid lubricant source. Figure 18 graphically represents the influence of graphite type on silicon

Figure 13 Optical micrographs showing solid lubricant buildup from Union Carbide CJPS carbon-graphite cage on support ball.
Test Conditions: 1000°F (538°C) for 49.2 hours (66 million stress cycles).



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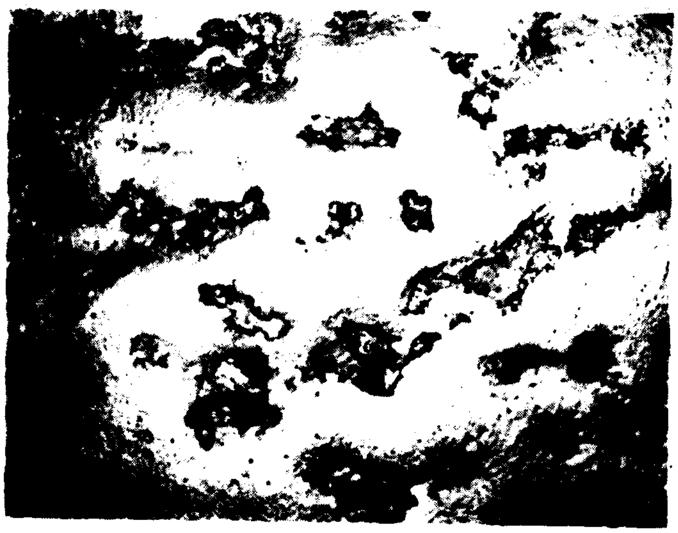
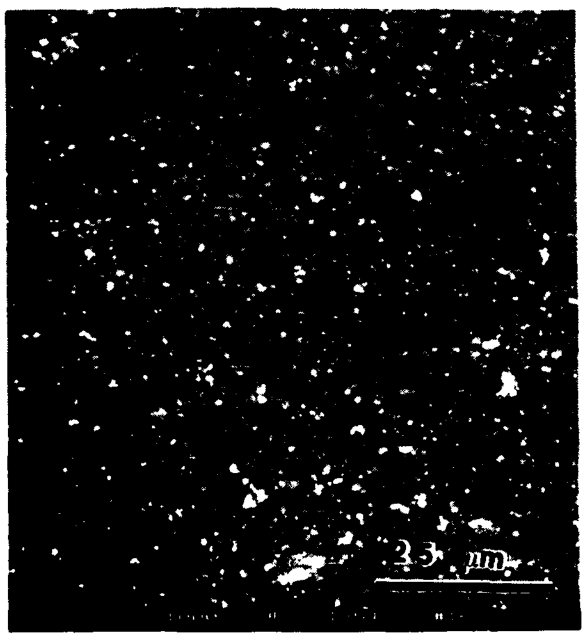


500x

Figure 14 Microfissures observed in Si_3N_4 spindle ball wear track from Test 16. A Union Carbide CJPS carbon-graphite cage was used as the solid lubricant source. Test conditions: 1000°F (538°C) for 49.2 hours (66 million stress cycles).

A. Optical 100x



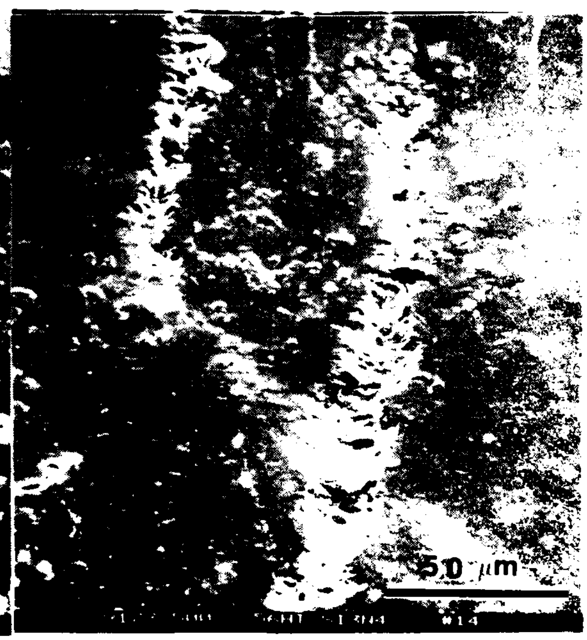
B. SEM

Figure 15 Microfissures observed in Si_3N_4 spindle ball wear track from Test 14. A Pure Carbon Co. 56HT graphite cage was used as the solid lubricant source test conditions: 1000°F (538°C) for 130.7 hours (176 million stress cycles).

A. Optical 100x

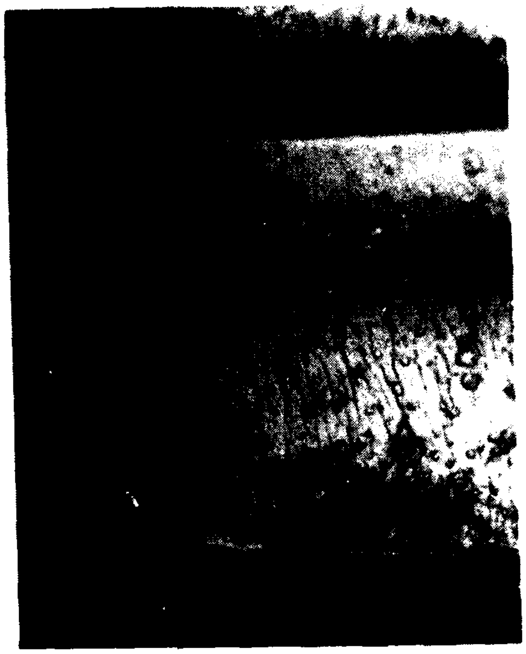
B. SEM

C. SEM

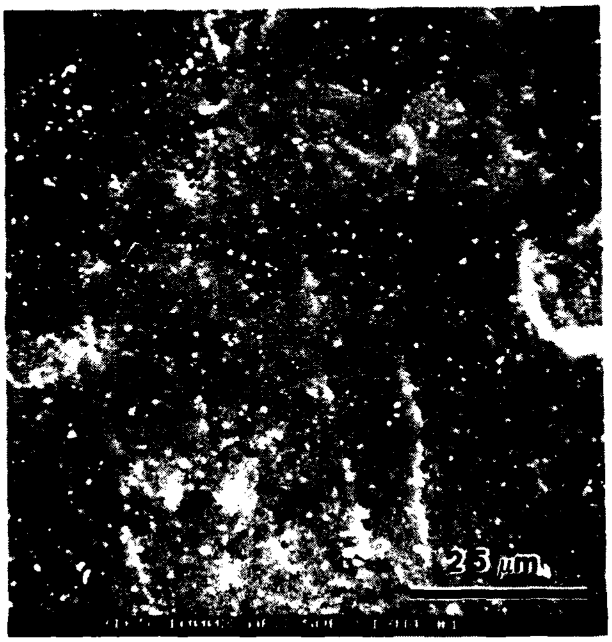


D. SEM

Figure 16 Microfissures observed in Si_3N_4 spindle ball wear track from Test 18. A POCO Graphite AFX-5QE graphite cage was used as the solid lubricant source. Test conditions: 1000°F (538°C) for 71.2 hours (96 million stress cycles).

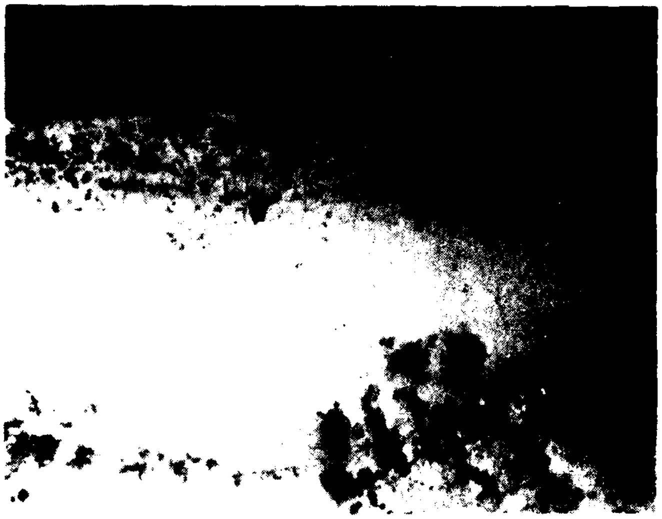


A. Optical 100 x

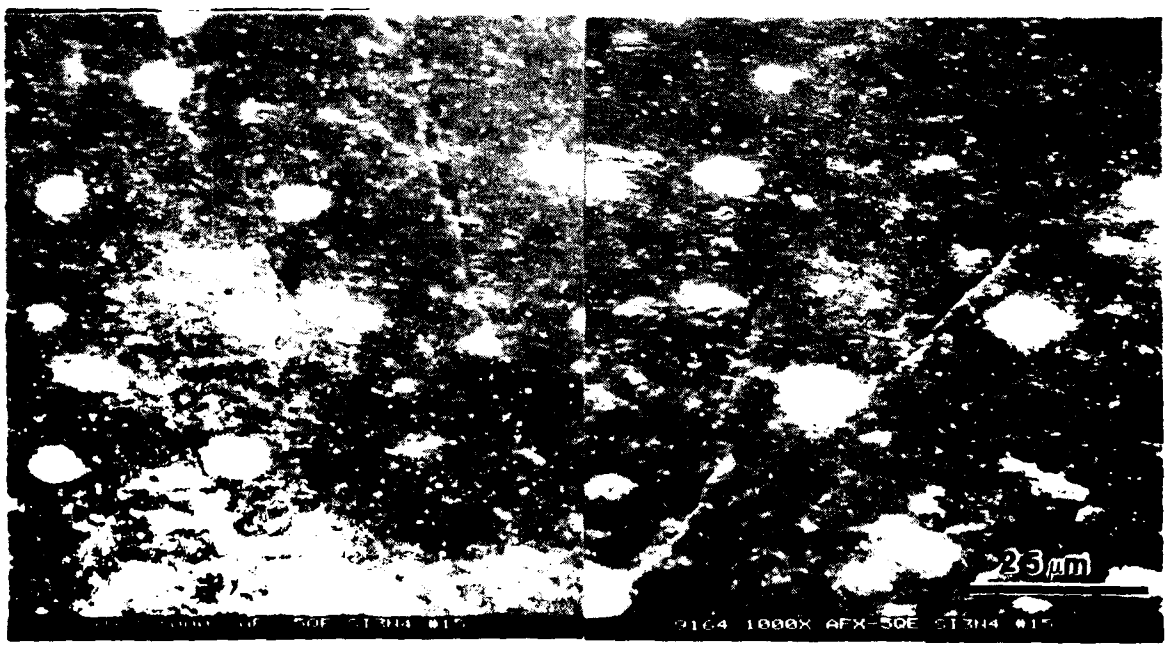


B. SEM

Figure 17 Microfissures observed in Si_3N_4 [illegible] ball wear track from Test 1[illegible]. A POCO Graphite AFX-5QE graphite cage was used as the solid lubricant source. Test conditions: 1000°F (538°C) for [illegible] hours (68 million stress cycles).

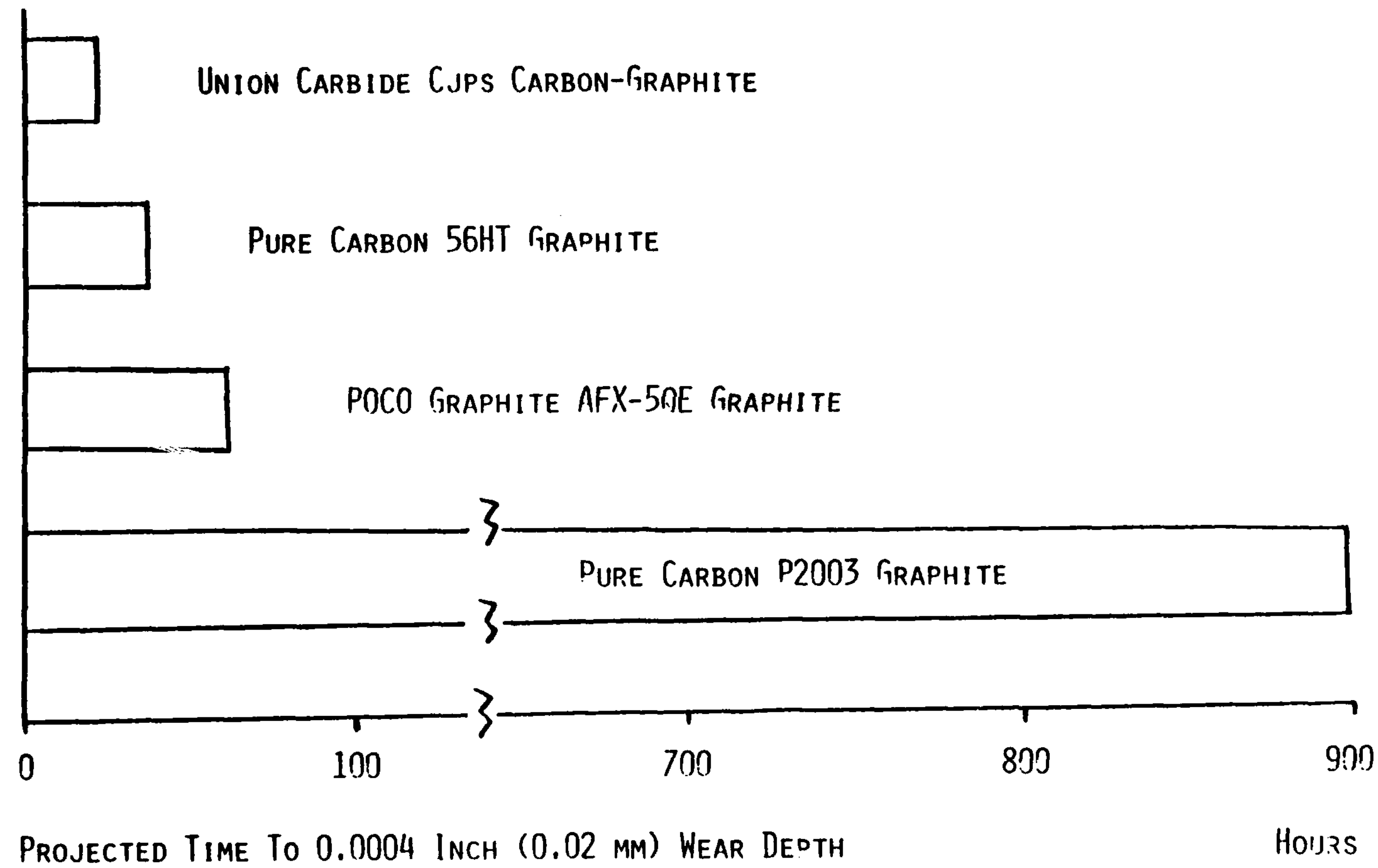


A. Optical 100x



B. SEM

Figure 18 Effect of various graphite cages on silicon nitride wear.
Test Conditions: 10,000 RPM, 1000°F (538°C), 400 ksi (2760 MPa).



nitride performance. The wear equation presented in Section 5.4 was used to estimate the test time required to produce a 0.0004 in (0.02 mm) deep track on the silicon nitride spindle ball.

It was believed that the proprietary impregnants were responsible for differences in the graphite film's ability to reduce silicon nitride wear. Some proprietary impregnants were evidently more successful than others at reducing oxidation of the thin graphite films on the silicon nitride surface. The reduction in graphite oxidation allows the graphite to shear along the basal planes in the graphite crystal structure. Graphite microstructure did not seem to have a significant effect on the test results. This result is not unexpected since the crystal structure was identical for all the graphite grades evaluated even though the microstructure or grain size was different.

Pure Carbon P2003 graphite resulted in the lowest wear coefficients, least surface distress, and the longest successful tests. Tests conducted for over 250 hours (348 million stress cycles) at 400 ksi (2760 MPa) contact stresses indicate that solid lubricated, silicon nitride bearings are feasible.

6.2.3.4 Silicon Nitride Surface Modifications

Silicon nitride spindle balls nitrided at Garrett had a dull, gray appearance in contrast to the dark black shiny surface of a typical bearing ball. One nitrided ball was tested in Test 21. Unfortunately, a used Pure Carbon P2003 cage had be to used for this test. The nitriding did not appear to significantly influence the silicon nitride wear rate. However, test results were complicated due to the use of previously tested graphite cages. No further testing was conducted using these silicon nitride balls.

6.2.4 1200°F (649°C) Tests

6.2.4.1 Graphite

Three tests were conducted at 1200°F with graphite cages. Pure Carbon P2003 and POCO AFX-5QE graphites were selected for these tests. The P2003 graphite was chosen due to the very low wear coefficients obtained at the 1000°F test temperatures. Although the POCO Graphite did not prevent silicon nitride wear as well as the P2003 graphite, the POCO Graphite AFX-5QE was evaluated due to its outstanding oxidation resistance at lower temperatures.

Pure Carbon P2003 was evaluated in Tests 17 and 20. These tests revealed that the silicon nitride wear coefficient was low,

approximately 1.5 x 10^{-6}, but that the solid lubricant transfer film was unstable. Figure 19 shows the charred solid lubricant build up on a silicon nitride spindle ball. Visual inspection of this cage revealed pores through which the impregnant had exuded.

A second test of silicon nitride at 1200°F (649°C) resulted in a continual increase of bearing noise as a function of time. Visual inspection revealed that the solid lubricant transfer was uneven resulting in a "wash board" surface on the spindle ball. The silicon nitride surface appearance was excellent. These tests reveal that an improved solid lubricant would be necessary to run continually at 1200°F (649°C).

Test No. 22 was conducted at 1200°F utilizing a POCO Graphite AFX-5QE cage. The 1200°F test resulted in considerable plastic flow and microspalling of the silicon nitride spindle ball wear track. Figure 20 shows a scanning electron photomicrograph of the wear track from this test.

6.3 Appearance of Solid Lubricant Cages

Figures 21 and 22 show graphite cages before and after four ball testing at 1000°F. The Pure Carbon P2003 graphite had a noticeable white, fluffy surface after high temperature testing. The other graphite materials evaluated did not appear to be altered.

Only one graphite based cage fractured. Figure 22 shows the Union Carbide CJPS carbon-graphite cage which fractured from the ball pocket. Fracture was believed to be related to a heavy layer of solid lubricant transferred from the cage to the support ball surface. This lubricant build up would decrease the clearance between the cage and support balls resulting in high stresses in the graphite cage and cage fracture.

The Westinghouse WSe_2-In-Ga cages fractured in a manner similar to the Union Carbide CJPS graphite. The solid lubricant build up caused these cages to fracture in a plane parallel to the plane containing the outer race. Fracture occurred in all ball pockets in the WSe_2-In-Ga composite cage.

Figure 23 shows an SEM photomicrograph of a P2003 ball pocket after 258 hours at 1000°F. X-ray spectrography detected a higher carbon concentration in the ball pockets, compared to the edge of the ball pocket. The debris at the edge of the ball contact had a greater proportion of magnesium, zinc, and phosphorus.

Figure 24 documents the cage wear locations typically observed during the 1000°F tests. The graphite cages demonstrated

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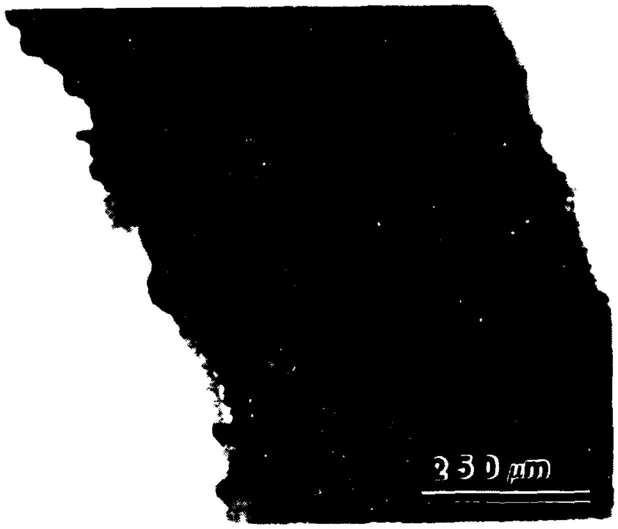
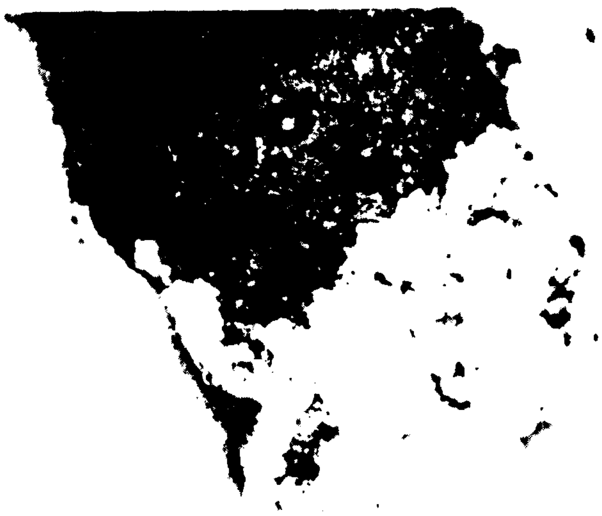
Figure 19 Optical micrographs showing charred graphite coating from Pure Carbon P2003 graphite on spindle ball after high temperature tests.
Test Conditions: 1200°F (649°C) for 23.8 hours (32.3 million stress cycles).



A. Wear Track 100x



B. Coating Partially Removed 100x

Figure 20 SEM photomicrographs of silicon nitride plastic flow and microspalling observed during high temperature test with a [illegible] Graphite AFX-5QE Cage. Test Conditions: 1200°F (649°C) for 29.8 hours (40 million stress cycles).

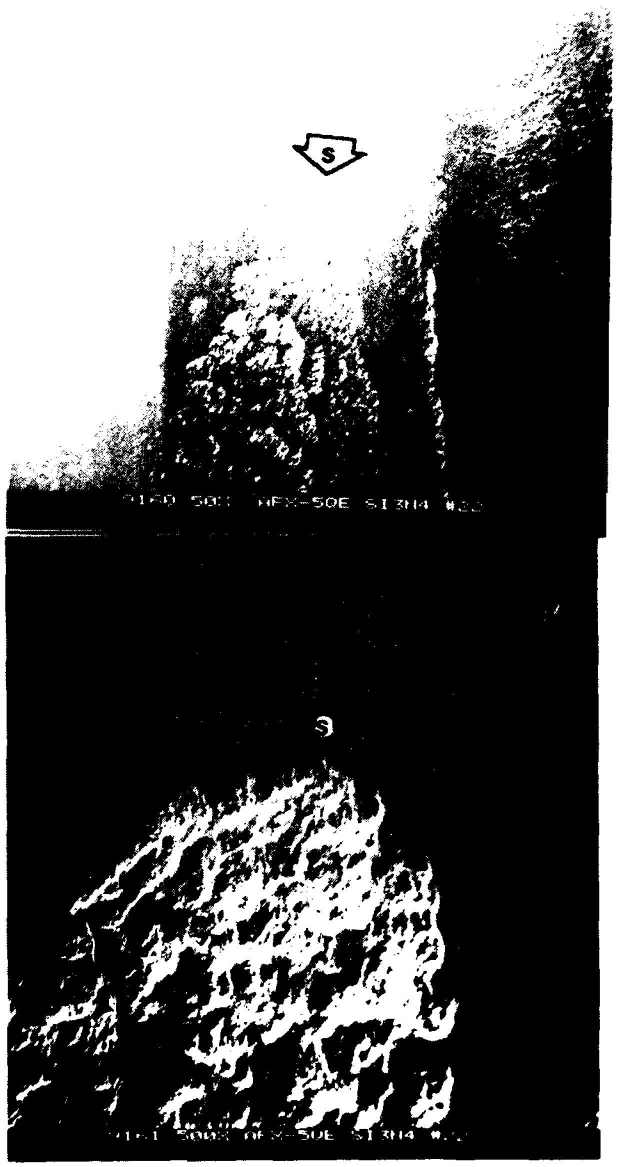
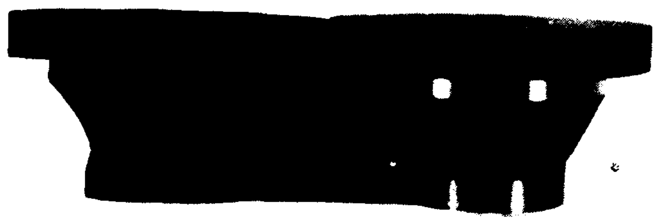


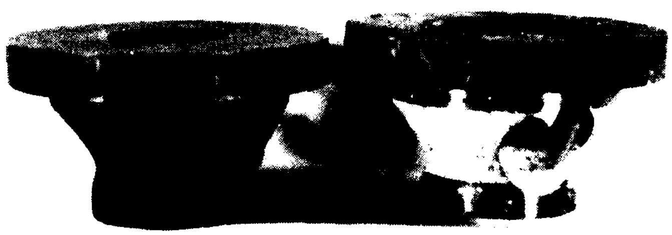
Figure 21 Photographs of L-23745 cage manufactured from Pure Carbon Co. 56HT and P2003 graphites before and after test.



PURE CARBON
56HT
(NEW)

PURE CARBON
56HT
(50 HOURS, 1000°F)

INCHES



PURE CARBON
P2003 GRAPHITE
(NEW)

PURE CARBON
P2003 GRAPHITE
(258 HOURS, 1000°F)

INCHES

Figure 22 Photographs of L-23745 cage manufactured from POCO Graphite, Inc. AFX-5QE graphite and Union Carbide Co. CJPS graphite-carbon before and after test.

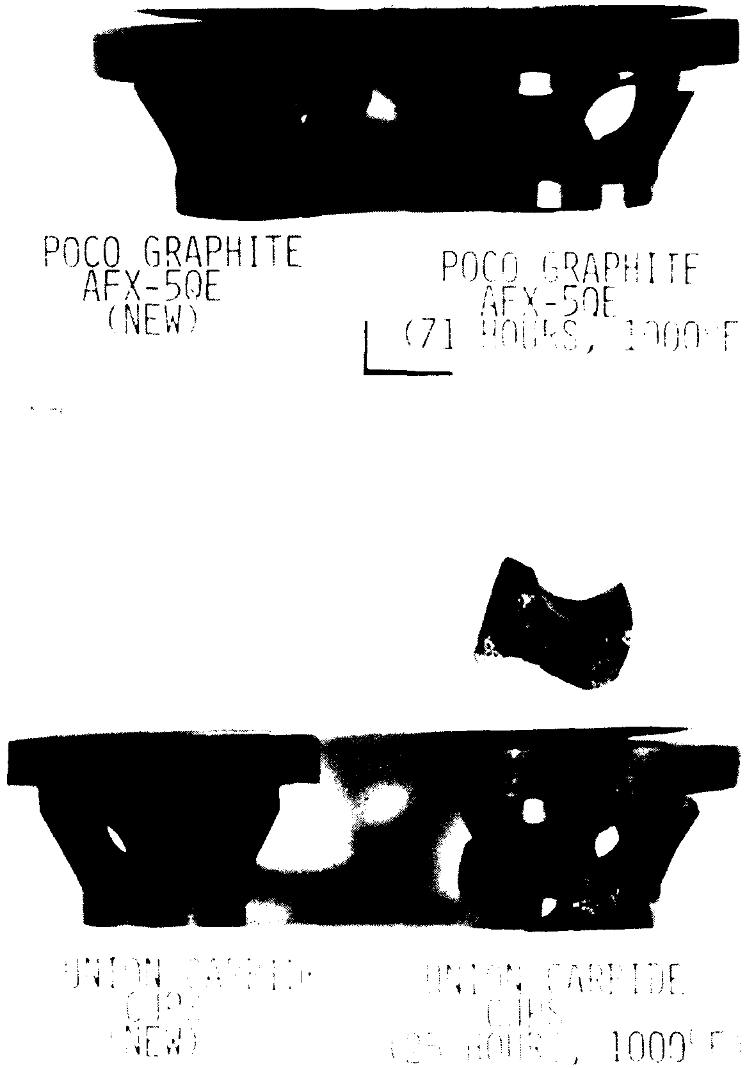
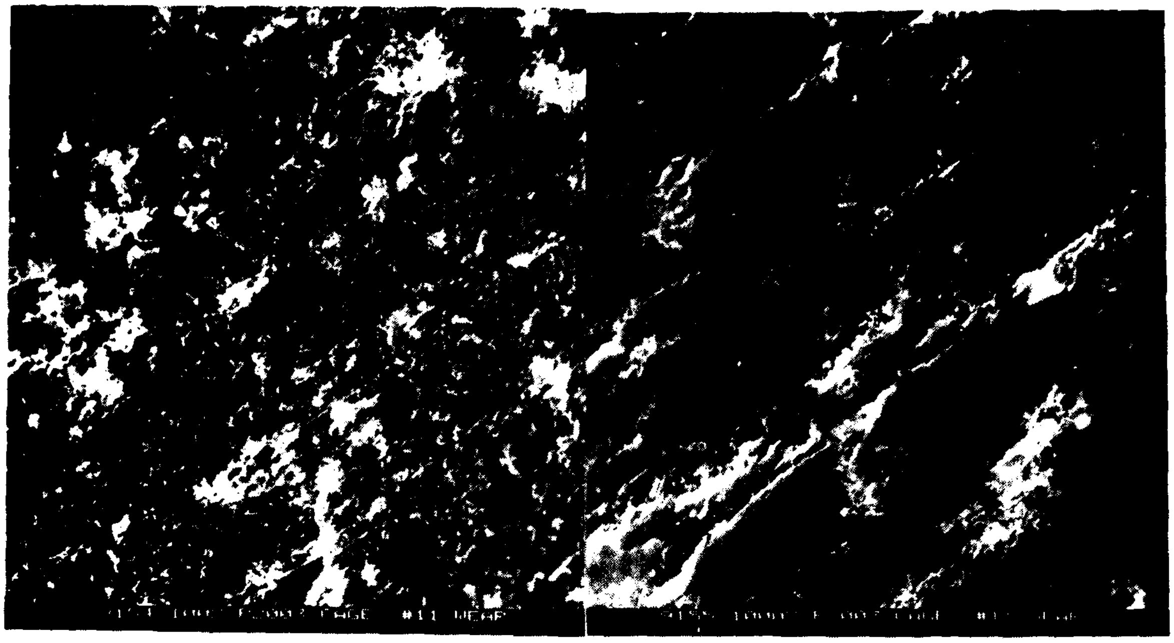


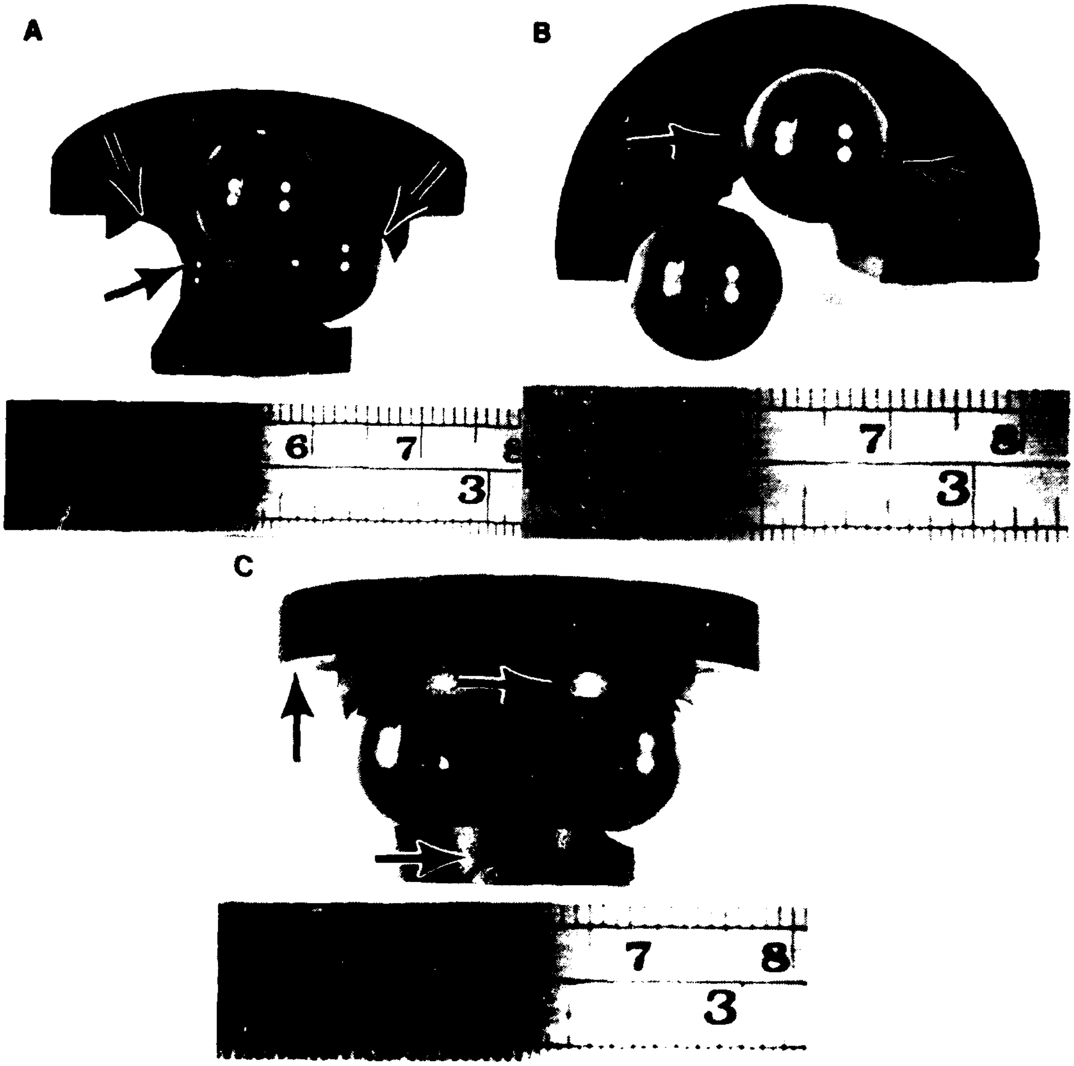
Figure 23 P2003 graphite cage surfaces after 258 hours (348 million stress cycles) at 1000°F (538°C).



A. Graphite surface at silicon nitride ball - graphite cage contact.

B. Graphite debris at edge of ball contact.

Figure 24 Photographs documenting cage wear locations in Pure Carbon 56HT graphite. Wear observed in this 56HT graphite cage was typical of all graphite based cages tested during the NAVAIR program. Arrows indicate wear locations.



consistent pattern of wear. Cage wear primarily occurred in the ball pocket as shown in Figure 24A and B. Initial cage wear was rapid at the cylindrical (cage) - spherical (ball) interface. The initial high wear rate assisted the solid lubricant transfer to support and spindle balls. As test time increased, the wear of the graphite cage pockets resulted in the cage pocket conforming to the silicon nitride ball surface. This increase in conformity reduces the stress on the graphite and the graphite wear rate. Later tests were conducted utilizing a used P2003 graphite cage. These tests produced a significantly higher wear coefficient at the 600°F and 1000°F test temperatures. Table 8 contains a comparison of silicon nitride wear coefficients in new and used Pure Carbon P2003 graphite.

Although initial graphite wear was rapid, once the cage pocket conformed to the ball surface, cage pocket wear appeared to decrease. This result may have been fortuitous due to a piloting of the cage in the areas depicted by arrows in Figure 24C. Cage designs for high speed, solid lubricated bearings will undoubtedly have to have a large ball to cage contact area in addition to a positive piloting mechanism.

6.4 Shock Pulse Analysis

An SKF Mark IV shock pulse analyzer was used to monitor frictional or kinetic energy generated by the four ball assembly as a funcion of temperature for a limited number of tests. Figure 25 summarizes the data recorded by this technique. Note that the values presented do not represent system vibration. For example, the WSe_2-In-Ga composite fractured at high temperature resulting in considerable rig vibration. Excessive vibration resulted in three vibration switch shut-offs of this test prior to test termination, but the kinetic energy remained at a low value.

As can be seen in Figure 25, oil lubrication and grease lubrication resulted in count rates of 80 and 20, respectively. The lower kinetic energy level of the grease was believed to be due to a dampening of the cage-cup impacts. Note the low values of kinetic energy recorded when a new Pure Carbon 56HT cage was used. The energy recorded over the temperature range 70°F to 1000°F was comparable to grease lubricated silicon nitride.

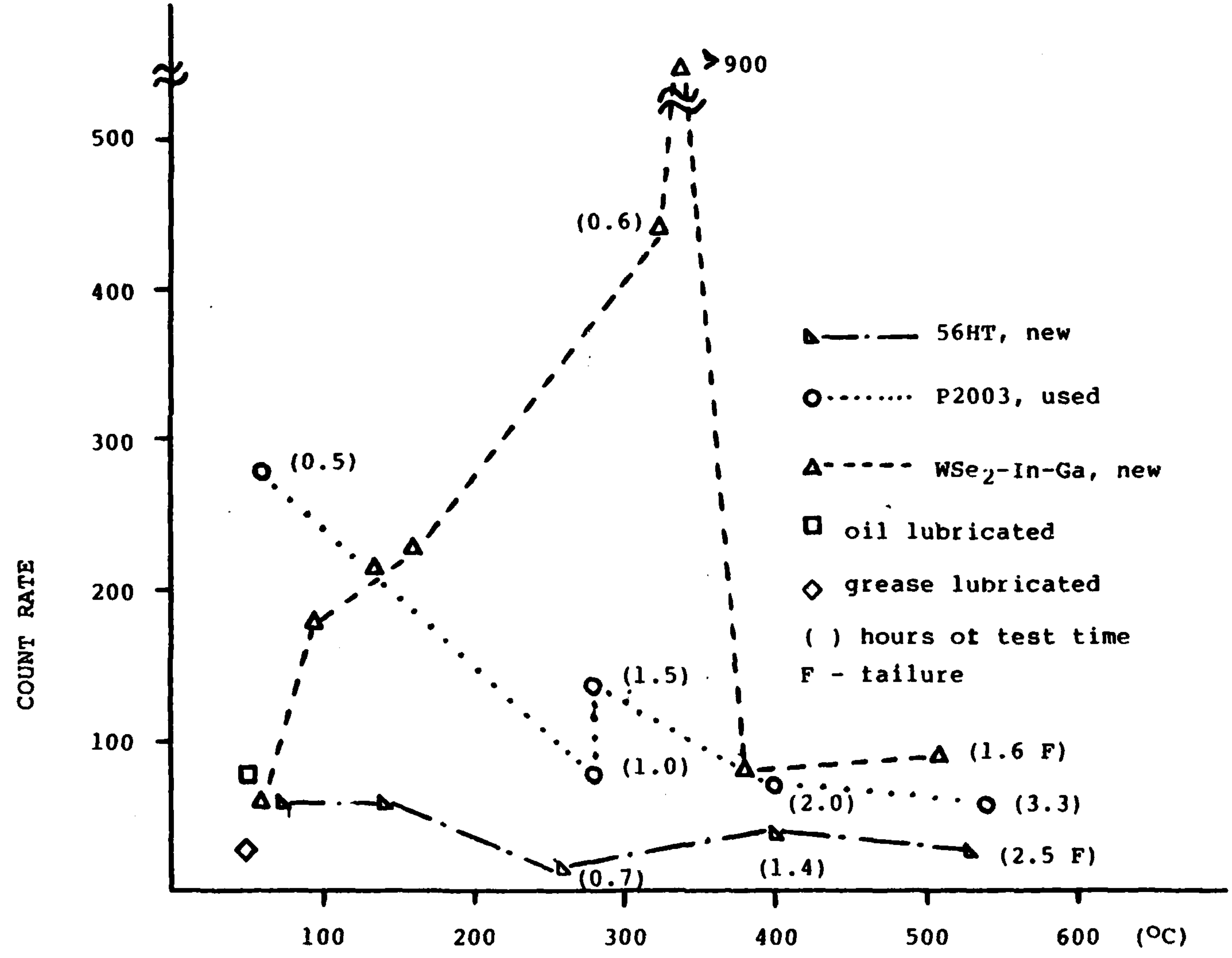
The shock pulse count rate for Westinghouse composite cage dramatically changed in the temperature range 100°F to 750°F. This result indicates that the Westinghouse composite has marginal lubricating ability. Perhaps the composite would offer some lubrication to silicon nitride above 750°F. However, it never produced the low kinetic or frictional level of the Pure Carbon 56HT or P2003 materials. The P2003 cage may have had a lower count rate had a new cage been available for test.

Table 8

Wear Rates of Silicon Nitride Obtained with "New" and "Used" Pure Carbon P2003 Graphite Cages

Test	Cage Condition	Temp. ($^{\circ}F$)	Hours	Stress Cycles ($\times 10^6$)	Wear Volume (mm^3)	Wear Coefficient ($\times 10^{-7}$)
9	New	1000	61	82	0.03	7
11	New	1000	258	348	0.07	4
12	New	1000	139	187	0.02	2
13	New	1000	256	345	0.04	2
23	Used in Test 6	600	54	73	0.129	34
24	Used in Tests 9 and 21	1000	12.3	16	0.47	540

Figure 25 Shock pulse count rate observed with solid and liquid lubrication during short term four ball tests.



The data obtained from limited shock pulse monitoring indicates the silicon nitride contacts tested with lubricating graphite transfer films dissipates kinetic energy at a rate similar to oil lubricated silicon nitride. Monitoring the count rate as the four ball test was initiated revealed that lubricant transfer was nearly instantaneous. Further studies would be necessary to relate lubricant breakdown with the kinetic energy generated.

7.0 Summary

Successful transfer of solid lubricant films from a cage to silicon nitride balls was demonstrated by rolling contact four ball tests. Tests at 1000°F, initiated at ambient temperature, revealed that transferred graphite films could provide lubrication to the silicon nitride balls over a wide temperature range. The lowest silicon nitride wear rates were achieved using Pure Carbon Co. P2003 graphite as the lubricant source. Tests conducted with this graphite for over 250 hours demonstrated the feasibility of high temperature solid lubricated silicon nitride bearings. Additional tests indicated that graphite-silicon nitride bearing systems can withstand short term temperature excursions as high as 1200°F.

Extensive optical and SEM examinations detected microfissuring of some silicon nitride surface. Microfissuring could occur at 1200°F and lower temperatures with inadequate lubrication. It should be noted that the test conditions were considered slightly accelerated due to the high contact stress utilized 400 ksi (2760 MPa).

Limited testing with used graphite cages resulted in slightly higher silicon nitride wear rates. The higher wear rates were believed to be related to increased silicon nitride ball--cage pocket conformity associated with the used cages. It appears that an initial high graphite wear rate is required to obtain very low silicon nitride wear rates, unless races and balls are precoated with a solid lubricant film compatible with the cage material.

This investigation revealed that silicon nitride has considerable potential as a high temperature bearing material, but the silicon nitride capabilities are not boundless. Therefore, extreme care must be taken in selecting bearing design, cage design, and the solid lubricant system for any high temperature bearing applications.

8.0 Recommendations

1. Future studies should concentrate on functionally testing solid lubricated silicon nitride bearings at high speeds and temperatures. It is recommended that initial rig tests be

conducted using hybrid bearings (steel rings and silicon nitride balls) to establish solid lubricant/cage design practices. High temperature bearing tests with silicon nitride bearings would be required to demonstrate successful silicon nitride bearing design and mounting practices.

2. After performance of a high speed, high temperature bearing has been demonstrated on a rig, solid lubricated silicon nitride bearings should be engine tested. Data available from rig tests should make it possible to "fine tune" a solid lubricated silicon nitride bearing for specific engine locations.

3. Further investigation of solid lubricant stability, solid lubricant systems, bearing design, and bearing mounting design will be necessary in order to obtain successful results in a wide range of propulsion engine applications. A goal of these studies should be to establish a framework for solid lubricated rolling contact design and application.

9. References

1. F. R. Morrison, J. Pirvics, and T. Yonushonis, "Establishment of Engineering Design Data for Hybrid Steel/Ceramic Ball Bearings," Naval Air Systems Command, Contract No. N00019-78-C-0304, SKF Report No. AL79T033 (1979).

2. F. R. Morrison, and T. Yonushonis, "Establishment of Engineering Design Data for Hybrid Steel/Ceramic Ball Bearings: Phase II," Naval Air Systems Command, Contract No. N00019-79-C-0418, SKF Report No. AT80T042, (1980).

3. J. W. Lucek, L. B. Sibley, and J. W. Rosenlieb, "Severe Environment Testing of Silicon Nitride Rolling Elements," Naval Air Systems Command, Contract N00019-77-C-0551, (1979).

4. L. C. Lipp, "Solid Lubricants - Their Advantages and Limitations," ASLE Lubrication Engineering, 32, [11], 574-584.

5. H. E. Sliney, "High Temperature Solid Lubricants; Part 1: Layer Lattice Compounds and Graphite," Mechanical Engineering, [2], (1974), 18-22.

6. H. E. Sliney, "High Temperature Solid Lubricants' Part 2: Polymers and Fluoride Coatings and Composites," Mechanical Engineering, [3], (1974), 34- 39.

7. M. B. Peterson, "High Temperature Lubrication," presented at the Symposium on Lubrication and Wear, University of Houston, (1963).

8. A. J. Haltner, "The Development and Evaluation of Lubricating Systems for Reusable Space Vehicles," prepared for NASA under Contract NAS-8-26292 (1973).

9. K. M. Taylor, L. B. Sibley, and J. C. Lawrence, "Development of a Ceramic Rolling Contact Bearing for High Temperature Use," Wear 6 (1963), 226- 240.

10. D. J. Boes and J. S. Cunningham, "The Effects of Design Parameters and Lubricants on the Performance of Solid Lubricated, High Speed--High Temperature Bearings," Westinghouse Research Laboratories Scientific Paper No. 68-9B-5-LUBER-P1 (1968).

11. H. E. Sliney, "Wide Temperature Spectrum Self Lubricating Coatings Prepared by Plasma Spraying," Thin Solid Films, 64 (1979), 211-217.

12. R. M. Gruver, and H. P. Kirchner, "Effect of Leached Surface Layers on Impact Damage and Remaining Strength of Silicon Nitride," 59 (1976), 85-86.

13. G. E. Gazza, H. Kock, and G. D. Quinn, "Hot Pressed Si_3N_4 with Improved Thermal Stability," American Ceramics Society Bulletin, 57 (1978), 1059-60.

14. Dr. Jim Hirvonen, Naval Research Laboratories, personal communication.

15. J. F. Archard and W. Hirst, "The Wear of Metals Under Unlubricated Conditions," Proc. Roy. Soc., Series A., 236 (1956).

16. SKF Industries, Inc., Technology Data on Mark IV Shock Pulse Analyzer, (1980).

17. D. Board, "Machinery Diagnosis Through Shock Pulse Monitoring," SKF Industries, Inc.

18. M. N. Gardos, "Solid Lubricated Rolling Element Bearings," Contract No. F33615-78-C-5196, DARPA Order No. 3576 by Hughes Aircraft Division, February (1980).

19. F. P. Bowden and D. Tabor, "The Friction and Lubrication of Solids, Clarendon Press (Oxford), (1950).

20. N. P. Suh, "The Delamination Theory of Wear," Wear 25 (1973), 111-124.

Appendix A

Norton Raw Material Certification Data

Powder Lot HN-15

Billets 1280 → 1285, 1292 → 1297, and 1307

Table A-1

X-Ray Radiographic Results and Density of Norton NC132 Hot Pressed Silicon Nitride

Billet Number	Density	Norton X-Ray No. *
1280	3.28	MMR-G21-33
1281	3.29	MMR-G21-33
1282	3.29	MMR-G21-33
1283	3.30	AG-B20909
1284	3.26	AG-B20909
1285	3.27	AG-B20909
1292	3.27	AG-B20917
1293	3.26	AG-B20917
1294	3.26	AG-B20917
1295	3.26	MMR-G30-3
1296	3.26	MMR-G30-3
1297	3.27	MMR-G30-3
1307	3.26	MMR-G37-6

$\bar{x}$ = 3.27

S.D. = 0.01

* Radiographic inspection showed no abnormalities.

Table A-2

Certification of Powder Lot HN-15 - Chemical Analysis

Element	Percent
Mg	0.94
Ca	0.03
Fe	0.29
Al	0.20
O_2	3.70
W	2.40

Table A-3
Certification of Powder Lot HN15 - Mechanical Properties

A. Room Temperature Four Point Flexure Strength

ksi	(MPa)
135.4	(933.6)
134.3	(926.0)
132.5	(913.6)
131.9	(909.5)
128.5	(886.0)
107.4	(740.5)
106.8	(736.4)
105.7	(728.8)
$\bar{x}$ = 122.8	$\bar{x}$ = (846.7)
S.D. = 13.6	S.D. = (93.8)

B. 2500°F (1370°C) Three Point Flexure Strength

ksi	(MPa)
47.6	(328)
46.9	(323)
45.4	(313)
44.6	(308)
$\bar{x}$ = 46.1	$\bar{x}$ = (318)
S.D. = 1.4	S.D. = (10)

Appendix B

Load Required To Produce A 400 ksi (2760 MPa) Contact Stress

Calculation of the Load Required to Produce a 400 Ksi (2760 MPa) Hertz Stress for 0.5 Inch (12.7 mm) Diameter Silicon Nitride Balls

From Timshenko and Goodier (1) the contact radius for two balls in contact which have the same elastic properties is described by:

$$a = \text{contact radius} = \sqrt[3]{\frac{3}{2} P \frac{R_1 \cdot R_2}{R_1 + R_2} \frac{1 - \nu^2}{E}} \quad (i)$$

The following parameters are known for the test conditions and the silicon nitride balls in contact and are defined.

Hertz Stress	=	q	=	400 Ksi (2758 MPa)
Ball Radius	=	R_1, R_2	=	0.25 in. (6.35 mm)
Poisson's Ratio	=	ν	=	0.25
Elastic Modulus	=	E	=	45×10^6 psi (310 Gpa) at 1000°F

Substituting into (i) we find the following:

$$a = 1.575 \times 10^{-3} \; P^{1/3} \quad (ii)$$

The maximum contact pressure is ralated to the load by the following equation:

$$q = \frac{3}{2} \frac{P}{\pi a^2} \quad (iii)$$

Substituting (ii) into (iii) yields the required load for a contact pressure q = 400 Ksi.

$$P = 8.97 \text{ pounds} \quad (39.9 \text{ N})$$

The vertical force, Q_4, required on the test ball for a four ball test will be:

$$Q_4 = 3 P \sin 44^\circ = 18.7 \text{ pounds} \quad (83.2 \text{ N})$$

with five ball test

$$Q_5 = 4 P \sin 44^\circ = 25 \text{ pounds} \quad (111 \text{ N})$$

Reference: S. Timoshenko and J. N. Goodier, Theory of Elasticity, McGraw-Hill Book Company, Inc. 1951.

Appendix C

Test Summaries 1 through 30

TEST NO. 1:

Ball Complement:	Four support balls, one spindle ball.
Spindle:	Zirconia rod with slot to turn spindle ball.
Spindle Run Out:	0.002 inch (0.05 mm) no load condition.
Test Temperature:	Ambient.
Lubrication:	None.
Test Summary:	Test initiated at room temperature to check out rig dynamics at 10,000 rpm. During first shut off of the rig after 30 seconds of test a severe vibration was noted. A second start up of the rig resulted in fracture of the zirconia rod ending the test.
Action:	Recheck system alignment.

TEST NO. 2:

Ball Complement:	Four support balls, one spindle ball.
Spindle:	Zirconia rod with slot to turn spindle ball.
Spindle Run Out:	Spindle run out was 0.001 inches (0.025 mm) under a no load condition. Checking the zirconia rod at a 25 pound (111 N) loading approximately 400 ksi (2760 MPa) contact stress at the silicon nitride ball resulted in a 0.008 inch (0.2 mm) spindle run out. The base plate containing the silicon nitride cup was shifted to yield a 0.002 inch (0.05 mm) run out at the zirconia/silicon nitride ball interface.
Test Temperature:	1000°F (538°C).
Lubrication:	None.
Test Summary:	The motor driving the spindle ball was started after the silicon nitride cup, support balls, and spindle balls had been stabilized at 1000°F. The assembly rotated for approximately 10 seconds before a switch shut off the

(Test No. 2 - continued)

driving motor. Disassembling the silicon nitride five ball assembly revealed considerable zirconia dust on the ball surfaces and fibers from the alumina insulation surrounding the metal housing.

Action: Recheck system alignment with zirconia spindle. Manufacture metal plate to retain alumina fibers and keep the fibers out of the silicon nitride bearing assembly.

TEST NO. 3:

Ball Complement: Four support balls, one spindle ball.

Spindle: Zirconia spindle, then metal taper.

Spindle Run Out: Spindle run out was checked with the zirconia rod in the unloaded condition and the loaded condition. Analysis of the data from the dial gauges indicated that the zirconia shaft was not concentric with silicon nitride spindle ball. Further checks of the spindle run out indicated that the metal-zirconia rod interface had a 0.006 inch (0.15 mm) run out when the zirconia-silicon nitride was aligned to 0.002 in (0.005 mm). Due to the problems in alignment encountered with the zirconia shaft used to drive the silicon nitride ball and the generation of abrasive particles from the zirconia shaft, a tapered metal shaft was used to drive the silicon nitride spindle ball. Room temperature checks of the run out and vibration indicated that the system was acceptable.

Test Temperature: 1000°F (538°C).

Lubrication: None.

Test Summary: The test was initiated at room temperature at 10,000 rpm. A 1000°F test temperature in the housing was obtained after 27 minutes of operation. The assembly was shut off due to vibration after 31 minutes of test. Disassembly of the full complement silicon nitride bearing revealed major wear of the

(Test No. 3 - continued)

spindle ball, support balls, and cup. Considerable fine silicon nitride debris was noted on the support balls and in the cup.

Action: Analyze debris, initiate next test with the first available solid lubricant cages.

Tests No. 4 through 30: Information Common to All Tests

Ball Complement: Three support balls, one spindle ball.

Spindle and Spindle Run Out: Tapered metal shaft, approximately 0.001 in. (0.025 mm).

TEST NO. 4:

Test Temperature: Goal was 1000°F (538°C), Actual 950°F (510°C).

Lubrication: Westinghouse Corp. WSe_2-In-Ga composite, Dicronite WS_2 solid film on balls.

Test Summary: Test was initiated at room temperature with the spindle speed slowly increased to 7500 rpm. The system operated smoothly as the temperature was increased to 950°F over a 32 minute period. At 950°F the noise from the test system increased dramatically. The test was stopped to avoid damaging the silicon nitride cup. Inspection of the balls revealed that the WS_2 film had disappeared. Little wear of the spindle ball was observed.

TEST NO. 5:

Test Temperature: Goal was 750°F (400°C), Actual 670°F (354°C).

Lubrication: Westinghouse Corp. WSe_2-In-Ga composite, Dicronite WS_2 solid film on balls.

Test Summary: Test was initiated at room temperature with the spindle speed slowly increased to 7500 rpm. The test temperature was slowly increased to 670°F (354°C) over a 17 minute period. At 670°F the

(Test No. 5 - continued)

noise and vibration increased which resulted in the vibra switch shutting off the test apparatus. Inspection revealed that an oxidation film had surrounded the support balls resulting in seizure of the balls in the cage.

TEST NO. 6:

Test Temperature: 1000°F (538°C).

Lubrication: Pure Carbon Co. P2003 graphite, E/M Lubricant's Microseal 100-1 on balls.

Test Summary: Test initiated at room temperature with the spindle speed increased to 10,000 rpm over a 5 minute period. Temperature was increased to 1000°F over a 34 minute period. The combination of solid lubricants and silicon nitride ran 34 hours before a vibra switch stopped the test. Two wear tracks were noted on the spindle ball. First successful test at 1000°F.

TEST NO.7:

Test Temperature: Pure Carbon Co. 56HT graphite, E/M Lubricant's Microseal 100-1 on balls.

Test Summary: Test initiated at room temperature and 10,000 rpm. Temperature was increased to 1000°F (538°C) over a 50 minute period. Forty hours of testing accomplished prior to vibra switch shut off. Optical analysis indicated that the balls were in good condition.

TEST NO.8:

Test Temperature: 1000°F (538°C).

Lubrication: Pure Carbon Co. 56HT graphite.

Test Summary: Test terminated after 50.4 hours. All components in excellent condition.

TEST NO. 9:

Test Temperature: $1000^{\circ}F$ ($538^{\circ}C$)

Lubrication: Pure Carbon Co. P2003 graphite.

Test Summary: Test terminated after 60.9 hours. All components in excellent condition.

TEST NO. 10:

Test Temperature: $1000^{\circ}F$ ($538^{\circ}C$).

Lubrication: Westinghouse WSe_2-In-Ga composite.

Test Summary: Cage hand polished to avoid cage seizing on support balls. The test assembly was heated to $1000^{\circ}F$, then the spindle was rotated by hand to apply a solid lubricant film. Spindle shut off five times at low speed (5000 rpm) due to vibration. After slowly increasing speed to 10,000 rpm "smoke" was emanating from the assembly. The system was allowed to run for 5 minutes prior to shut down. Disassembly of the system revealed the Westinghouse composite cage had fractured. A considerable build up of solid lubricant was observed on the spindle and support balls.

TEST NO. 11:

Test Temperature and Time: $1000^{\circ}F$ ($538^{\circ}C$) for 258 hours.

Lubrication: Pure Carbon Co. P2003.

Test Summary: Test terminated after 258 hours with all components in excellent condition. Very small wear track observed on spindle ball.

TEST NO. 12:

Test Temperature and Time: $1000^{\circ}F$ ($538^{\circ}C$) for 139 hours.

Lubrication: Pure Carbon Co. P2003.

(Test No. 12 - continued)

Test Summary: Test terminated after 139 hours with all components in excellent condition.

TEST NO. 13:

Test Temperature and Time: 1000°F (538°C) for 256 hours.

Lubrication: Pure Carbon Co. P2003.

Test Summary: Test terminated after 256 hours with all components in excellent condition. Very small wear track observed on the spindle ball.

TEST NO. 14:

Test Temperature and Time: 1000°F (538°C) for 130.7 hours.

Lubrication: Pure Carbon 56HT.

Test Summary: Test ran uneventfully for 130.7 hours when the vibraswitch shut off the test. The test goal was 250 hours. Visual inspection indicated that iron oxide from the metal housing had contaminated the test system. The silicon nitride balls were highly polished and the spindle ball wear track was considerably greater than observed when a Pure Carbon P2003 cage has been used. However, due to iron oxide contamination the results of this test are considered inconclusive. Further long term test evaluations (greater than 50 hours) and all 1200°F will be conducted in rig 213 which has a stainless steel housing.

TEST NO. 15:

Test Temperature and Time: 1000°F (538°C) for 50.5 hours.

Lubrication: POCO Graphite AFX-5QE.

Test Summary: Test terminated after 50.5 hours with all components in excellent condition. Visual examination of the wear track

(Test No. 15 - continued)

indicates considerably more wear of silicon nitride when lubricated by POCO Graphite AFX-5QE compared to Pure Carbon P2003.

TEST NO. 16:

Test Temperature and Time:	1000°F (538°C) for 49.2 hours.
Lubrication:	Union Carbide CJPS.
Test Summary:	Test terminated after 49.2 hours. Visual examination indicated that the silicon nitride balls were heavily coated with a solid lubricant film. The silicon nitride support balls exhibited strong evidence of tracking in the silicon nitride cup. Although a relatively "thick" solid lubricant coated the support balls, visual examination indicates that the wear rate with a Union Carbide CJPS cage was considerably greater than the wear rate observed with a Pure Carbon P2003 cage.

TEST NO. 17:

Test Temperature and Time:	1000°F (538°C) for 17 hours and 1200°F (649°C) for 7 hours.
Lubrication:	Pure Carbon Co. P2003.
Test Summary:	Increase temperature from 200°F to 1200°F over a 49 minute period. The test system was held at 1200°F for a two hour period, then the temperature was reduced to 1000°F for 17 hours (over night). The temperature was then increased to 1200°F for an additional 5 hours of operation. Disassembly of the test system indicated that all components were in excellent condition. Tracking of support balls was evident. A solid lubricant film had transferred to the spindle ball. Visual inspection revealed that very little wear of the spindle ball had occurred. Solid lubricant film build-up may result in problems with clearance in a bearing.

TEST NO. 18:

Test Temperature and Time:	1000°F (538°C) for 71.2 hours.
Lubrication:	POCO Graphite AFX-5QE.
Test Summary:	Support balls had a slightly mottled surface appearance due to transfer of solid lubricant. Visual inspection of the spindle ball wear track indicates a higher wear rate with POCO Graphite AFX-5QE than Pure Carbon P2003.

TEST NO. 19:

Test Temperature and Time:	1000°F (538°C) for 25.3 hours.
Lubrication:	Union Carbide CJPS.
Test Summary:	Vibraswitch shut the test system off after 25 hours at 1000°F. Visual examination of support and spindle balls revealed heavy wear and delamination of the silicon nitride. It appears as though the CJPS cage reacted with the silicon nitride at the 1000°F test temperature.

TEST NO. 20:

Test Temperature and Time:	1200°F (649°C) for 6.9 hours.
Lubrication:	Pure Carbon Co. P2003.
Test Summary:	Attained 1200°F test temperature in 45 minutes. Vibration level appeared to increase throughout the test. Testing was terminated after 6.9 hours due to vibration level. An appreciable uneven build up of solid lubricant was noted on the spindle ball.

TEST NO. 21:

Test Temperature and Time:	1000°F (538°C) for 49.8 hours.

(Test No. 21 - continued)

Lubrication:	Pure Carbon Co. P2003 cage used previously in Test No. 9.
Test Summary:	An AiResearch nitrided spindle ball was used for this test. Wear of the spindle ball was greater than typically observed with a new P2003 cage.

TEST NO. 22:

Test Temperature and Time:	1200°F (649°C) for 29.8 hours.
Lubrication:	POCO Graphite AFX-5QE.
Test Summary:	Considerable noise was noted during test start up, although the housing vibration appeared to be normal. The spindle ball had a wide wear track and exhibited considerable pitting. General condition of spindle ball was poor.

TEST NO. 23:

Test Temperature and Time:	600°F (315°C) for 54 hours.
Lubrication:	Pure Carbon Co. P2003 cage used previously in Test No. 6.
Test Summary:	Test ran uneventfully at 600°F for 54 hours. Spindle ball wear visually greater at 600°F than at 1000°F. Increased wear rate may be due to cage pocket-ball conformity due to the utilization of a cage tested in Test No. 6.

TEST NO. 24:

Test Temperature and Time:	1000°F (538°C) for 12.3 hours.
Lubrication:	Pure Carbon Co. P2003 cage used previously in Test No. 9 and 21.
Test Summary:	Vibration switch shut off the system after 12.3 hours. Spindle ball wear track was uneven and considerably

(Test No. 24 - continued)

deeper than 250 hour test conducted using a new P2003 cage. Support balls had a dull finish. One support ball was gouged.

TEST NO. 25:

Test Temperature and Time:	970°F (520°C) maximum temperature, 25 minutes total test time.
Lubrication:	MoS_2 on all support and spindle balls - five ball test - no cage.
Test Summary:	Test temperature reached 970°F prior to increase in vibration and noise level. The system was shut off by the operator. MoS_2 coating appeared to be completely worn off the support balls. Considerable wear of spindle ball observed.

TEST NO. 26:

Test Temperature and Time:	Incrementally increased from room temperature to 1000°F (538°C), 2.5 hour test.
Lubrication:	Pure Carbon Co. 56HT.
Test Summary:	Vibration shut off system after 2.5 hours. Shock pulse analyzer was used to monitor kinetic and frictional energy. Kinetic energy was similar to oil lubricated silicon nitride contacts.

TEST NO. 27:

Test Temperature and Time:	Incrementally increased temperature from room temperature to 1000°F (538°C), 3.3 hour test.
Lubrication:	Pure Carbon Co. P2003 cage, used previously.
Test Summary:	Shock pulse analyzer was used to monitor energy levels as a function of temperature. Energy recorded was similar to oil lubricated silicon nitride.

TEST NO. 28:

Test Temperature and Time:	Incrementally increased temperature from room temperature to 1000°F (538°C), 1.6 hour test.
Lubrication:	Westinghouse Corp. WSe_2-In-Ga composite.
Test Summary:	Shock pulse analyzer used to monitor kinetic events. Kinetic energy level increased dramatically as temperature was increased from 200°F to 750°F. A significant drop in energy recorded occured at 750°F. Vibration shut off the test system three times at temperatures greater than 900°F. Disassembly of the test system revealed the WSe_2-In-Ga cage had fractured.

TEST NO. 29:

Test Temperature and Time:	Room temperature, 10 minutes.
Lubrication:	DTE light oil.
Test Summary:	Established baseline reading of 80 on shock pulse analyzer for silicon nitride with copious oil lubrication. A graphite cage was used for this test.

TEST NO. 30:

Test Temperature and Time:	Room temperature, 10 minutes.
Lubrication:	Grease.
Test Summary:	Grease lubrication evidently dampened kinetic energy associated with the cage. Established baseline reading of 10 to 20 on shock pulse analyzer for grease lubrication. A graphite cage was used for this test.

Appendix D

Shock Pulse Analyzer

Kinetic and frictional energy dissipated by the silicon nitride simulated bearing assembly was monitored using an SKF Mark IV Analyzer [17]. This equipment was used on a limited basis to determine the effect of solid lubricant films on the kinetic energy generated by the silicon nitride contacts.

High frequency vibration analysis or shock pulse analysis utilizes the transducer resonant output at frequencies several orders of magnitude above the dynamic "operating frequency" that makes the shock pulse technique insensitive to background vibration, but highly sensitive to the kinetic and frictional energy generated by defects in rotating machinery.

Shock pulse analysis operates on the principle that discrepant parts within a machine (such as pitted, spalled, brinnelled, or skidding bearing elements) release abnormal amounts of frictional and/or kinetic energy. In the frequency domain, friction generates "white" noise (nearly constant amplitude at all frequencies up to several hundred KHz) and a kinetic event, by virtue of its very short rise time, generates only very gradually decaying amplitudes as a function of increasing frequency. The harmonic amplitudes of machine vibration, however, decay more rapidly with increasing frequency. Thus, above a certain frequency, f_E, the amplitude of frictional and kinetic energy release will be greater than that of machine vibration.

Additionally, a piezoelectric accelerometer has a resonant frequency that is typically greater than f_E, so that it can amplify the kinetic and frictional energy it detects when mounted on a machine's housing. A bandpass filter on the accelerometer output, screens out transducer response at all frequencies significantly above or below the sensor's resonant frequency. This means that the time domain amplitude modulations of the bandpass filter output are almost entirely a function of kinetic and frictional energy fluctuations in the machine. An envelope detector follows the amplitude modulations of the bandpass filter output, thus providing a "Shock Pulse" each time the accelerometer is resonated by the release of kinetic and frictional energy.

This shock pulse signal can be analyzed in several ways; the two most effective of which are Shock Pulse Spectral Analysis and Shock Profile Area Measurement. In Shock Pulse Spectral Analysis the shock pulse train is fed into a conventional narrow band spectrum analyzer which performs a Fourier transformation and displays the relative amplitude of shock pulses as a function of the frequency at which they occur. In Shock Profile Area Measurement, the shock pulses are analyzed to construct a curve of shock pulse rate as a function of the shock pulse absolute amplitude. This curve is called a "Shock Profile," and the area

under the curve, which is proportional to the total kinetic/frictional energy release within the machine, is called the "Shock Profile Area" (SPA). Thus the SPA is a diagnostic parameter capable of detecting discrepant parts whose failure modes generate abnormal levels of kinetic and frictional energy. The shock pulse profile area was used to monitor the effects of the solid lubricant film during this program.

The effects on test operation of selected solid lubricant cages and solid lubricant coatings were monitored by the SPA technique as a function of temperature. Test time was short, one to two hours, in order to allow investigation of a number of different solid lubricant systems. At ambient temperature, shock pulse measurements of grease and oil lubricated silicon nitride were obtained for comparison to solid lubricated silicon nitride contacts.

The piezoelectric transducer was attached to the base plate supporting the housing as shown in Figure 4.

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